

FINAL UPSTREAM PATRICK BAYOU CHARACTERIZATION
SAMPLING AND ANALYSIS PLAN
PATRICK BAYOU SUPERFUND SITE
REMEDIAL INVESTIGATION
DEER PARK, TEXAS

# **Prepared for**

Patrick Bayou Joint Defense Group

# **Prepared by**

Anchor QEA, LLC 614 Magnolia Avenue Ocean Springs, Mississippi 39564

June 2011

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Attachment 2	Field Forms
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# **LIST OF ACRONYMS AND ABBREVIATIONS**

Abbreviation	Definition
°C	degrees Celsius
°F	degrees Fahrenheit
ng/L	nanograms per liter
μg/L	micrograms per liter

Anchor QEA Anchor QEA, LLC

AOC Administrative Order on Consent

Cs-137 Cesium-137 cm centimeter

COC chain-of-custody

COPC chemical of potential concern

DGPS differential global positioning system

GIS Geographical Information System

GPS global positioning system
HASP Health and Safety Plan
HDPE high density polyethylene
HSC Houston Ship Channel

JDG Joint Defense Group

mg/kg milligrams per kilogram

NAD83 North American Datum 1983

PAH polycyclic aromatic hydrocarbons

PBUC Patrick Bayou Upstream Characterization

Pb-210 Lead-210

PCB polychlorinated biphenyl

POC point of contact

PPE personal protective equipment
PQL practical quantitation limits

PSCR Preliminary Site Characterization Report

PTFE polytetrafluoroethylene

QA/QC quality assurance/quality control

QAP Quality Assurance Plan

QAPP Quality Assurance Project Plan

RI/FS Remedial Investigation/Feasibility Study

SAP Sampling and Analysis Plan

SH State Highway

Site Patrick Bayou Superfund Site
SOP Standard Operating Procedure
SVOC semi-volatile organic compounds

TCEQ Texas Commission on Environmental Quality

TxDOT Texas Department of Transportation
USEPA U.S. Environmental Protection Agency

VOC volatile organic compounds
WWTP Waste Water Treatment Plant

#### 1 INTRODUCTION AND PURPOSE

This Sampling and Analysis Plan (SAP) describes the rationale, objectives, study design, and methods for the characterization of the sediments in the upstream portion of the Patrick Bayou Superfund Site (Site; Stations PB066 to PB101), and culverts that run underneath State Highway (SH) 225, just upstream of the Site boundary. The SAP will be conducted by the Patrick Bayou Joint Defense Group (JDG) as part of the Remedial Investigation/Feasibility Study (RI/FS) for the Site, required in the Administrative Order on Consent (AOC) and Settlement Agreement with the U.S. Environmental Protection Agency (USEPA) dated January 31, 2006. The RI Work Plan (Anchor 2007a) provides a general framework for Site remedial investigations that is based on an adaptive management process, whereby work is completed, results are evaluated, the understanding of the Site updated, and future work plans are developed and revised as appropriate. The order of future work is prioritized so that existing and new data are complementary and leveraged towards building a better conceptual understanding of the Site. In this process, work occurs in phases, and each phase of work is fully described in either work plans or SAPs for USEPA review and approval prior to initiation.

The proposed data collection activities in this SAP were developed based on review of the results of the Site-wide sediment and water column sampling in 2009, which identified concentrations of polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) in the sediments at Station PB081, and PCBs in surface water at Station PB076.

### 1.1 Project Management

The JDG retained Anchor QEA, LLC (Anchor QEA) to perform the RI. A comprehensive description of the project organization, schedule, and contacts is provided in the Quality Assurance Project Plan (QAPP; submitted as part of the RI Work Plan; Anchor 2007a). For this study, Anchor QEA will conduct the field work and data analysis. Table 1 provides an updated list of names and Quality Assurance (QA) responsibilities of key project personnel who will be involved in sampling and analysis activities described in this SAP.

#### 1.2 Site Description

Patrick Bayou is a tributary of the Houston Ship Channel (HSC) in Harris County, Texas (Figure 1). Patrick Bayou discharges into the south side of the HSC approximately 2.3 miles upstream of its confluence with the San Jacinto River. The Site itself and its physical features are described in more detail in the *Preliminary Site Characterization Report* (PSCR; Anchor 2006), and the *Work Package 2 Hydrodynamic Field Data Collection and Contaminant Source Evaluation Data Report* (Work Package 2 Data Report; Anchor 2007b); a brief summary is provided in the following paragraphs.

The Site originates north of SH 225 downstream of the City of Deer Park Waste Water Treatment Plant (WWTP) and flows north approximately 10,200 feet to the HSC (Figures 2a through 2c and Figure 3). The drainage upstream of the Site originates in the City of Deer Park and consists of trapezoidal, concrete-lined ditches, which transition into large culverts underneath SH 225. These culverts emerge into a gunite-lined channel at the upstream Site boundary. The gunite-lined portion of the Site extends approximately 1,800 feet north (downstream), to a more natural channel bottom, bordered by natural and armored banks, that make up the remainder of the Site. A tributary of Patrick Bayou, referred to as the East Fork, joins the Site near Bayou Station PB065 of the main channel (Figures 2c and 3). The East Fork is a small intermittent stream, approximately 5,500 feet long, that flows in a northwesterly direction.

Patrick Bayou widens to approximately 330 feet at the confluence of the East Fork tributary and the main channel and ranges between 40 to 480 feet wide for the next 2,500 feet (between Bayou Station PB065 and PB040). Narrower parts of Patrick Bayou are associated with structures (bridges) and channel modifications. Two small islands approximately 0.35 acre in size are located near the center of the channel, before Patrick Bayou makes an easterly turn between Bayou Stations PB020 and PB015. Approximately 340 feet downstream of the islands, an elevated pipeline and bridge cross Patrick Bayou to a loading terminal, located on the HSC. Patrick Bayou discharges into the HSC approximately 1,000 feet from the crossing and bridge. This lower portion of Patrick Bayou varies between 300 and 500 feet, reaching its widest point where it joins the HSC.

#### 2 PREVIOUS RESULTS, STUDY OBJECTIVES, AND DESIGN

The 2009 evaluation of the concentration of chemicals of potential concern (COPCs) in sediments and surface waters at the Site were conducted for the following reasons (Anchor QEA 2010):

- To provide a synoptic, Site-wide understanding of the distribution of COPC concentrations in surface sediments that is representative of the mixing zone layer (0 to 10 centimeters [cm]; Anchor QEA 2009a).
- To provide a comprehensive and spatially representative characterization of the distribution of COPC concentrations, thus reducing uncertainty in the statistical analysis of surface sediment concentration estimates over the entire Site.
- To refine the surface water COPC selection list that was incomplete due to limited or no surface water data for dioxins, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), PCBs, and pesticides, and which represented a data gap in the selection of COPCs and exposure assessment for Site receptors.

The concentrations of PCBs and PAHs in the 2009 surface sediment characterization event at Station PB081 were 21.7 milligrams per kilogram (mg/kg) and 1,306 mg/kg, respectively. PAHs and other COPCs were not detected in surface waters just downstream of this area; however, the concentrations of PCBs at Station PB076 in the 2009 sampling event ranged between 58.0 nanograms per liter (ng/L) and 117 ng/L, using one-half of the detection limit for congeners that were below the analytical detection limit (Anchor QEA 2010).

Based on these observations, this SAP involves the collection of additional sediment and water quality data to develop a better understanding of the distribution of PCBs, PAHs, and other Site COPCs in upstream areas of the Site. The SAP is primarily focused on the evaluation of PCBs in this area because they are the only COPC identified in surface water besides pesticides, and because of their importance to ecological and human health risk evaluations.

#### 2.1 Sampling and Analysis Objectives

The objectives of this SAP include:

- Establish the extent of PCBs, PAHs, and other COPCs at upstream locations.
- Investigate the sediment condition and vertical composition of sediments present in the culverts at SH 225 to determine if historic and/or ongoing discharges associated with the local watershed drainages have, or are, contributing to COPC loading at the Site<sup>1</sup>.
- Fill in data gaps in water column sampling between Stations PB066 and PB101. Past water column sampling only involved collecting samples at Station PB076 in this area of the Site. Because detectable concentrations of PCBs were found at this station, and it is the most upstream station where PCBs were found in surface water, additional data are required to more closely define the source of surface water PCBs.

#### 2.2 Relationship to Other Activities

Previous evaluations at the Site that are relevant to this SAP have focused on determining the vertical distribution of potential contaminants; collecting hydrologic and hydrodynamic information; developing a better understanding of sediment physical characteristics (Anchor 2007b); developing integrated Site watershed, hydrodynamic, and sediment transport models (Anchor QEA 2009b); defining the Site list of COPCs (Anchor 2008a); determining the depth of the sediment mixing zone at the Site (Anchor 2008b), and determining the distribution of COPCs in surface sediments and surface water (Anchor QEA 2010).

In the vertical characterization evaluation, sediment cores were collected at 14 stations using a 3-inch diameter piston core with a maximum penetration and recovery depth of approximately 8 feet. All cores were manually pushed to the full length of the core, or until refusal, whichever occurred first. Separate cores were collected at a subset of five stations for radiochemical analysis using Cesium-137 (Cs-137) and Lead-210 (Pb-210), in addition to the bulk chemistry cores. Each core was sectioned into various depth intervals for bulk sediment chemistry and radiochemistry analyses.

<sup>&</sup>lt;sup>1</sup> Although Patrick Bayou is a tidally influenced system, upstream areas (i.e. upstream of Station PB076) are generally not influenced by tides and flow is generally downstream. However, it should be noted that it is possible that tidal flow reversals may occur on an infrequent basis and thereby act as a source to upstream sediments as well.

In addition, five data collection platforms were utilized at key inflow and outflow points across the Site to collect the field hydrologic data, and sediment cores were collected at 12 locations for the Sedflume testing to aid in the development of the Site hydrologic and sediment transport models. The modeling framework for this study consisted of three models that are linked together: 1) watershed (hydrology); 2) hydrodynamics (EFDC); and 3) sediment transport (SEDZLJ). These models were developed to evaluate sediment stability during high-flow events and changes in the bulk COPC composition of surface sediments associated with sediment deposition during multi-year periods (Anchor QEA 2009b).

The effect of deposition of sediment from external sources on changes in sediment composition of the mixing-zone layer is being used to estimate the rate of changes in COPC concentrations in the Site sediment bed in the sediment transport model (Anchor QEA 2009b). Observations from the vertical coring investigations and sediment transport modeling indicate there is a significant amount of ongoing sedimentation at the Site involving a mixture of new sediment entering the Site and redistribution of on-site materials.

In addition to providing a base map showing the current distribution of COPCs in surface sediments over the entire Site, the current understanding of how ongoing sediment transport and deposition at the Site affects current and future potential ecological and human health risk posed by COPCs in surface sediments will be refined, based on the results of this investigation and the results of the 2009 Site-wide surface sediment and surface water characterization. This investigation is in essence an extension of the 2009 evaluation, and is designed to fill in data gaps that were identified during the review of the 2009 data.

#### 2.3 Sample Collection Design

The previous investigations in Patrick Bayou, described above, have shown that COPCs, including PCBs and PAHs, have historically accumulated in soft sediments. The upstream area that is the focus of this SAP generally has a higher hydraulic gradient compared to downstream areas of the Site, and in large part is characterized by a harder substrate with a large proportion of coarse sand, gravel, and cobble size materials. The materials are difficult to sample and past sampling results show that they generally have lower concentrations of PCBs (approximately 1 mg/kg to 5 mg/kg, compared to 21.7 mg/kg at PB081). However,

there are discrete areas of finer grained soft sediment accumulations, such as the area around Station PB081, that have exhibited elevated concentrations of PCBs and PAHs. The first upstream indication of detectable concentrations of PCBs in surface water in the 2009 sampling event was at Station PB076. These observations suggest that any additional characterization of the distribution of PCBs and other COPCs in upstream areas of the Site should focus on identifying areas of soft sediment accumulation. The activities associated with this investigation include the following:

- 1. Sediment probing to determine the location and thickness of soft sediment accumulations in the upstream areas of the Site between PB066 and PB101 (Figure 4).
- 2. Collecting sediment samples from soft sediment accumulations identified in the probing. Areas containing sediments that can be collected using a conventional mini-Ponar sampling apparatus will be sampled. These samples will be tested on-site for total PCBs using field immunoassay test kits for PCBs. The results of the immunoassay testing will be used to identify specific samples for additional laboratory analysis of the sediments. In addition, split samples will be retained and archived from each grab sample for potential future analysis (see Section 5.6.2).
- 3. Surface water sampling between Stations PB066 and PB101. Water samples will be collected at four locations, including one each at Stations PB066 and PB101 (Figure 4), and two additional, as yet unidentified, locations. The two as yet unidentified sample locations will coincide with areas where significant concentrations of PCBs are identified in the immunoassay testing.
- 4. Probing and sampling sediments in the culverts underneath SH 225. Based on past observations, there are significant accumulations of sediments in the easternmost culvert under SH 225. One core sample (assuming 3-foot depth) will be collected in the eastern culvert sediments and grab samples will be collected in each of the other four culverts. These samples will be analyzed at a laboratory off-site for Site COPCs.

#### 2.4 Documentation and Records

Complete and accurate records of data collection, data correction, and data analysis will be maintained. Integrity of this information will be maintained throughout all data transfers and manipulations. Procedures used to generate, transform, and validate data are critical for effective data management. A summary of the data management procedures are provided in

the RI Work Plan QAPP and Data Management Plan (Anchor 2007a) and are incorporated into this SAP by reference.

#### 3 METHODS AND PROCEDURES

# 3.1 Experimental Design – Sediment Probing and Surface Sediment Collection Station PB066 to PB101

The sediment probing and sediment collection portion of this investigation will occur in the upstream portion of Patrick Bayou between Stations PB066 and PB101 and in the box culverts below SH 225.

# 3.1.1 Sediment Probing Sample Locations and Frequency – Station PB066 to Station PB101

The bottom of the channel is visible in shallow portions of the study area between PB066 and PB101, and the initial field investigation will focus on visually identifying finer grained sediment accumulations in those shallow areas by walking the shoreline, or by inspections from a boat. Visually identified finer grained sediment accumulations will subsequently be probed using a metal rod, and sampled at locations that contain more than 10 cm of predominately sand-size and smaller materials. Global positioning system (GPS) coordinates will be recorded at each probing and sampling location.

In those areas where the bottom of the channel is not visible enough to sufficiently identify the texture of the substrate, probing will be conducted to identify the type of substrate that is present. Longitudinal and lateral probing transects in the channel will vary between 20- and 50-foot spacing depending on Site access, obstructions, and the heterogeneity of the substrate. More intensive probing will occur in areas where soft sediment accumulations are identified, and will be adjusted to delineate the horizontal and vertical extent of the accumulation. GPS coordinates will be recorded at each probing location.

Up to 36 grab samples will be collected from identified soft sediment accumulations that have a thickness of 10 cm or more, to determine the surficial concentrations of total PCBs using immunoassay test kits on-site. The locations of sediment sampling will be determined by the field team during the sediment probing, and will include sampling in accessible locations where accumulations of soft sediment are identified. Each sample will be split, and six of the split samples will be submitted for off-site laboratory analysis of PCB Aroclors and PAHs.

### 3.1.2 Target Analyte List – Stations PB066 to Station PB101

Thirty-six sediment grab samples will be collected to determine the surficial concentrations of total PCBs in the upstream areas between PB066 and PB101 using immunoassay test kits on-site (Table 2). Splits from 6 of the 36 grab samples collected will be submitted to an off-site laboratory for the following analyses:

- PCB Aroclors
- PAHs (identified in Table 4)

The field team has the flexibility to adjust the number of splits and/or grab samples as needed given findings during the sampling event<sup>2</sup>.

#### 3.2 Experimental Design – Box Culvert Sediment Sampling

Sediment samples will be collected at the upstream end of the box culverts that run underneath SH 225. These samples may be representative of potential historic and ongoing COPC loading from off-site upstream areas of the watershed.

An addendum to the Site Health and Safety Plan (HASP; Anchor QEA 2009c) has been prepared to support sediment sampling activities in the culverts to address potential confined space entry hazards. The HASP addendum is included as Attachment 1.

# 3.2.1 Sample Locations and Frequency - Box Culverts

One sediment core of up to 100 cm (approximately 3 feet in total length) will be collected in the eastern culvert and sampled in 33-cm (approximate 1-foot) intervals. One sediment surface sample consisting of the top 10 cm will be collected from each remaining four culverts.

<sup>&</sup>lt;sup>2</sup> Samples that show a range of relatively elevated Total PCB concentration from the immunoassay test will be selected for lab analysis. Only if the extent and existence of soft sediments is considerably limited between PB066 and PB101 (which is not expected, based on previous sampling events) would less than six samples be sent to the off-site lab for analysis. Upward adjustments may be considered based on field results and observations from the field team.

#### 3.2.2 Target Analyte List – Box Culverts

The sediment grab samples and core collected from the culverts at SH 225 will be submitted to a laboratory for the following analyses:

- Specific gravity
- Grain size
- Total organic carbon
- Metals (identified in Table 4)
- Mercury
- PAHs (identified in Table 4)
- PCB congeners (1 to 209)
- Dioxins and furans (identified in Table 4)

#### 3.3 Experimental Design – Surface Water

Surface water collection will be conducted 24 hours after sediment probing and sampling is complete, to minimize potential interferences from suspended particulate materials associated with sediment probing and collection. The surface water study design was created to meet the objectives of this SAP. Texas Commission on Environmental Quality (TCEQ) guidance for water collection (Chapter 5 in TCEQ 2003) was also consulted.

# 3.3.1 Sample Locations and Frequency – Surface Water

Four surface water samples will be collected from the upstream portion of Patrick Bayou between Stations PB066 and PB101. Two of the four samples will be collected at the upstream and downstream ends of the area of interest, with the two remaining sample locations identified by the field team based on a review of the field test results of the sediment sampling. The decision will be based on the area with the highest relative total PCB concentrations. One sample will be collected immediately upstream and one sample immediately downstream of the area with the elevated total PCB concentrations. Samples will be collected only during a slack or out-going tide. At the suggestion provided in TCEQ (2003) guidance, sampling will be delayed for at least 48 hours after a heavy rainfall event, to be defined as greater than 1 inch per 24-hour period for the purposes of this work.

## 3.3.2 Target Analyte List – Surface Water

Surface water samples collected during this task will be submitted to an analytical laboratory for analysis according to the sample design summary provided in Table 3. The analyses include:

- PCB congeners (1 to 209)
- Total organic carbon
- Total suspended solids

# 4 LABORATORY ANALYTICAL METHODS, QUALITY CONTROL, AND MEASUREMENT QUALITY OBJECTIVES

Analytical methods and the associated method reporting limits for sediment samples collected as part of this SAP are listed in Table 4. Analytical methods and target practical quantitation limits (PQL) for surface water samples are provided in Table 5.

A summary of laboratory quality control samples and frequency of analysis for sediment sampling are listed in Table 6 and for surface water sampling in Table 7.

Laboratory measurement quality objectives for precision, accuracy, and completeness of sediment and water column chemistry analysis for this SAP are listed in Tables 8 and 9, respectively.

#### 5 FIELD ACTIVITY METHODS AND PROCEDURES

Details regarding project organization, schedule, contacts, access, utility clearance, and equipment, are provided in this section.

#### 5.1 Project Organization, Schedule, and Contacts

A comprehensive description of the project organization, schedule, and contacts is provided in the QAPP (submitted as part of the RI Work Plan; Anchor 2007a). For this study, Anchor QEA will conduct the field work as described in Section 1.1 and Table 1.

The field investigation is currently expected to occur in May of 2011. This schedule is subject to change based on agency approval of this sampling plan, weather delays, and other factors that may be unavoidable.

#### 5.2 Access and Sampling Permission

The Site may be accessed through four privately owned facilities: Shell Oil – Deer Park Refining Services, Shell Chemical L.P. – Deer Park Chemical Plant, Lubrizol Corporation, and OxyVinyls L.P. The following personnel are facility-specific points of contact (POCs) that will oversee the field activities occurring at their respective facilities:

- Jeff Stevenson Shell Chemical Deer Park Chemical Plant
- Norman Mollard Lubrizol Corporation
- Jeff Adamski OxyVinyls L.P.

Sampling in the culverts under SH 225 will be accessed through City of Deer Park and Texas Department of Transportation (TxDOT) right-of-way areas. Anchor QEA will identify access and permit requirements prior to field crew mobilization, and coordinate with appropriate parties within the City of Deer Park and TxDOT concerning specific sampling dates after access agreements have been executed.

#### 5.3 Utility Clearances

The nature of the sampling activities is unlikely to create a hazard due to underground utilities or submerged pipelines; however, intrusive subsample sampling activities or

sediment probing will not be performed near any known or observable structures. Due to the industrial nature of the Site, it is not anticipated that any residential underground utilities will exist at the proposed sampling sites. Location of the underground utilities will be coordinated with industrial facilities located along the Site boundaries. If underground or submerged utilities are present at a specific sampling location, the sampling location will be moved to avoid the utility.

#### 5.4 Equipment, Supplies and Sampling Containers

The analytical laboratory will provide certified, pre-cleaned, USEPA-approved containers for all samples. Prior to shipping, the analytical laboratory will add preservative, where required, according to USEPA protocols. Sample containers for sediment and surface water are described in Tables 10 and 11, respectively.

Necessary equipment, supplies, and sampling containers will be shipped or carried to the Site. Equipment and supplies may be shipped directly to the field from the vendor or to Anchor QEA's Gulf Coast or Houston offices for inspection prior to deployment or use in the field at the Site. All equipment and supplies will be inspected and tested, as needed, prior to field use.

#### 5.5 Surveying

Horizontal positioning at each sampling location within the channel will be determined using a differential global positioning system (DGPS) with a handheld GPS unit as a backup if necessary. Station positions will be recorded in latitude and longitude to the nearest 0.01 second in the North American Datum 1983 (NAD83). The accuracy of the horizontal coordinates will be within 1 foot of the actual location based on typical DGPS functionality. The distance into the box culvert from the upstream opening will be measured to determine horizontal positioning at each sampling location within the box culverts.

## 5.6 Sample Collection Procedures

The following subsections describe the procedures for sediment probing, collecting surface sediment samples, and conducting sediment PCB field assays, collecting a sediment core, and

collecting surface water samples. The sample processing procedures for sediment collection and surface water collection are also detailed.

#### 5.6.1 Sediment Probing

The field crew will mobilize to survey the sediments via small boat and via shoreline access where boat access is limited to determine the extent, location, and depth of soft sediments. As discussed in Section 3.1.1, the bottom of the channel is visible in shallow portions of the study area between PB066 and PB101, and the initial field investigation will focus on visually identifying finer grained sediment accumulations in those shallow areas by walking the shoreline or by inspection from a boat. Visually identified finer grained sediment accumulations will subsequently be probed using a metal rod, and sampled at locations that contain more than 10 cm of predominately sand-sized and smaller materials. GPS coordinates will be recorded at each probing and sampling location.

In those areas where the bottom of the channel is not visible enough to sufficiently identify the texture of the substrate, probing will be conducted to identify the type of substrate that is present. Longitudinal and lateral probing transects in the channel will vary between 20- and 50-foot spacing depending on Site access, obstructions, and the heterogeneity of the substrate. More intensive probing will occur in areas where soft sediment accumulations are identified, and will be adjusted to delineate the horizontal and vertical extent of the accumulation. GPS coordinates will be recorded at each probing location.

A metal 3/8-inch steel rod, or equivalent, marked at 1-cm intervals will be used to probe sediment depths from Stations PB066 to PB101. The rod will be lowered to the sediment-water interface and water depth will be noted using the marked 1-cm intervals on the rod. The soft sediment depth will then be measured by pushing the rod into the sediment until refusal, using reasonable single-human force. The depth of the penetrated sediment will be noted by subtracting the water depth from the depth at which refusal was met. The following measurements will be noted on the sediment probing log form (Attachment 2):

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- Time and date
- Probing station identification
- Station coordinates (in latitude/longitude)

- Water depth (cm)
- Depth of refusal (cm)
- Sediment thickness (to the nearest cm)
- Estimated sediment type (i.e., muddy [cohesive] bed, sandy [non-cohesive] bed, rocky bed); based on the type of resistance met by the pole
- Any presence of sheen or other distinguishing characteristics observed on the water surface during performance of probing or on the probe tip after completing probing activities.

Additional notes for each station will be recorded in the field log book and photographs taken of any unusual conditions encountered (sheens or distinguishing characteristics observed during probing or on the probing tip). The probing rod will be deployed from the sampling vessel when the depth of water allows for the draft of the sampling vessel. In shallow areas (i.e., generally less than 12 inches of water) and areas inaccessible by the vessel, probing will be performed without the use of the vessel, provided safe access to the measuring location is practical. Care will be taken to probe in a location(s) that is undisturbed (e.g., not walked over) prior to probing. Any deviations to the above field probing procedure shall be noted by the field team lead, in coordination with the project manager.

# 5.6.2 Surface Sediment Sampling

A total of up to 36 grab samples will be collected and tested as described below from areas where soft sediment accumulation thicknesses are 10 cm or greater. These samples will be tested for total PCBs using immunoassay test kits on-site. Each sample will be split, and a portion preserved for possible future laboratory based analyses. Confirmation analysis by an off-site laboratory will be conducted on six split samples from the grab samples for Aroclor PCB analyses and select PAHs. All 36 samples will be retained on ice, pending results from the PCB assays. Split samples from six of the PCB assay samples with the highest measured total PCB concentrations will be included for confirmation analysis by the off-site laboratory. The remaining 30 samples will be archived for potential future analysis.

Sediment grab samples will also be collected from the sediments existing in the culverts at SH 225. One grab sample will be collected from each culvert, with the exception of the eastern culvert. Samples collected from the culverts will be analyzed for specific gravity, grain size, total organic carbon, metals, mercury, PAHs, PCB congeners, and dioxin and furans. The distance into the box culvert from the upstream opening will be measured to determine horizontal positioning at each sampling location within the box culverts.

The targeted sample depth will be from the surface of the sediment-water interface to 10 cm below the surface for sediment chemistry samples. A small Ekman dredge grab sampler, van Veen grab sampler, or similar device will be used for sediment collection. The grab sampler will be deployed and retrieved at a rate of approximately 1 foot per second to minimize contacting the bottom at an angle and potential disturbance of the sediment surface within the sampler.

Material collected with the sampling device will be evaluated by the field lead for acceptability using the following criteria:

- The sampler is not overfilled.
- Overlying water is present (may not be applicable to exposed intertidal sediment samples collected at low tide).
- The overlying water (if present) is not excessively turbid.
- The sediment surface is relatively undisturbed.
- An adequate penetration depth is attained (i.e., ideally to enable sampling of the undisturbed surface sediment).

If a sample fails to meet any of the above criteria, it will be rejected and discarded away from the station.

After a sediment sample is judged to be acceptable, any overlying water will be siphoned off and the upper 10 cm of sediment will be collected in accordance with USEPA (1997) guidelines. If a grab sampler is used, then decontaminated stainless-steel spoons will be used to collect the sediment from the grab sampler. A stainless-steel ruler will be used with all sampling devices to ensure that the sampling criterion for adequate penetration has been met and that the correct amount (i.e., 10 cm) of sediment has been removed.

Sample collection activities will begin at the most downstream location and proceed upstream to minimize any potential for sample interference caused by disturbed sediment.

#### 5.6.2.1 Surface Sediment Sample Processing

All working surfaces and instruments used in the processing area will be thoroughly cleaned, decontaminated, and covered with aluminum foil to minimize outside contamination between samples events (see Section 5.7). Disposable gloves will be discarded after processing each station and replaced prior to handling decontaminated instruments or work surfaces.

Sample containers will be kept in packaging as received from the analytical laboratory until use; a sample container will be withdrawn only when a sample is to be collected and will be returned to a cooler containing completed samples. Table 10 indicates holding times and preservatives.

The surface sediment samples collected in this sampling event will be processed as follows:

- The general description (including penetration and recovery) of the grab sampler will be recorded on the appropriate log form (Attachment 2).
- A stainless steel spoon will be used to remove sediment from the grab sampler. Care
  will be taken to not sample sediment that is in contact with the sample device by only
  collecting sediments from the center of sample device.
- The sediment will be placed into a stainless steel bowl and homogenized in the field. Samples will be homogenized prior to placing in the sample containers. The general procedure will consist of:
  - Removing unrepresentative materials (twigs, shells, leaves, etc.) and documenting in the appropriate field log
  - Quickly and efficiently mixing the sample
  - Stirring the sediment until texture, color, and moisture homogeneity is achieved
- Using a clean, stainless steel spoon, pre-labeled sample containers will be completely filled, as indicated in Table 10.
- Immediately after filling the sample containers with sediment, the screw cap will be placed on the sample container and tightened, wiping jar threads as necessary to

- ensure a tight seal.
- All sample containers will be thoroughly checked for proper identification, analysis type, and lid tightness.
- Each container will be packed carefully to prevent breakage and placed inside of a cooler with ice for storage at the proper temperature (4 ± 2 degrees Celsius [°C] for all samples).

#### 5.6.2.2 Surface Sediment PCB Field Assays

PCB field assays will be performed using the RaPID Assay Test Kit made by SDIX of Newark, Delaware. The RaPID Assay PCB Test Kit has a minimum detection limit of 0.5 ppm Total PCBs as Aroclor 1285 (see Attachment 3). A separate aliquot of each surface sediment sample will be collected in order to perform PCB field assays. Samples must be dried to less than 30 percent moisture content prior to extraction. Drying may occur using a gravity filter system, a vacuum assisted gravity filter system, or in a low temperature (less than 225 degrees Fahrenheit [°F]) oven. Testing will be accomplished on representative samples prior to the field program to determine the most appropriate drying technique. Samples will be extracted, prepared, and analyzed for PCB analyses as described in the Sample Extraction Kit User's Guide and the RaPID Assay Test Kit User's Guide which can be found in Attachment 3. The techniques are summarized as follows:

#### Extraction:

- Weigh out  $10 \pm 0.1$  grams of dried sample into an extraction jar.
- Add the entire contents of one ampule of solvent (20 milliliters [mL] of Methanol) and immediately cap jar to prevent evaporation.
- Shake the jar vigorously for one minute.
- Allow the sample to settle for one minute or until a liquid solvent layer is observed above the sample.
- Pipette the solvent layer into the bottom portion of the filtration unit and press the top portion of the filtration unit into the bottom portion until it snaps together or until the majority of the liquid has passed upward through the filter and place on a flat surface. Note: Do not store the sample extract in the filtrate unit for extended periods of time due to the possibility of evaporative loss.

- Dilute 25 microliters ( $\mu$ L) of the extracted sample into a pre-measured diluents vial from the extraction kit.
- Cap the vial and invert several times to mix.

Preparation: (note testing will be accomplished in an on-site indoor laboratory)

- Allow reagents to come to ambient temperature prior to analysis and warm up the spectrophotometer for at least 30 minutes.
- Remove nine clean, blank test tubes for standards and control and one test tube for each sample. Prepare the three standards and negative control in duplicate and the control and samples in singlicate.
- Separate the upper test tube rack from the lower magnetic base and place labeled test tubes into the rack.
- Add 200 μL of standards, control, or sample into the appropriate tubes.
- Add 250 µL of Enzyme conjugate down the inside wall of each tube.
- Thoroughly mix the magnetic particles and add 500 µL of magnetic particles to each tube. Vortex each tube on low speed for one to two seconds.
- Incubate for 15 minutes at room temperature.
- After incubation, combine the upper test tube rack with the magnetic base and press all tubes into the base. Allow two minutes for the particles to separate.
- Invert the combined rack assembly over a sink and pour out the contents of the tubes.
- With the rack inverted, gently blot the test tube rims on several layers of paper towels to remove as much liquid as possible. Do not bang the rack, as this may dislodge the magnetic particles and affect the results.
- Add 1 mL of the Washing Solution down the inside wall of each tube and vortex the tubes for one to two seconds.
- Press all tubes back into the base and wait two minutes.
- Pour out contents as described previously and repeat washing procedure.
- Remove upper test tube rack (with tubes) from the magnetic base; add 500  $\mu L$  of Color Reagent, and vortex for one to two seconds.
- Incubate the tubes for 20 minutes at room temperature and then add 500  $\mu$ L of Stop Solution.

#### Analysis:

- Results should be read within 15 minutes of the addition of the Stop Solution.
- Wipe the outside of each tube to remove fingerprints and smudges prior to analysis.
- Refer to the RaPID Assay PCB Test Kit User's Guide for photometer parameter settings.
- Follow the instrument prompts to read the absorbance of all tubes.
- Record the sample name, absorbance, dilution factor, and concentration of each sample on the analytical bench sheet.
- If the sample concentration is greater than the upper level of calibration, the sample must be diluted and reanalyzed.

#### 5.6.2.2.1 PCB Field Assay Performance

In 1998, the USEPA published an Environmental Technology Verification Report which evaluated the performance of the RaPID Assay System for PCB Analysis (USEPA, 1998). This report found that the RaPID's worst-case precision (25% RSD) was comparable to the best-case precision (21% RSD) of the reference laboratory. The report also characterized the RaPID's accuracy as slightly biased, with both positive and negative biases that were not reflected in the overall accuracy. The average percent recovery of the performance evaluation samples analyzed using the RaPID Assay System was 103%. The report concluded that the RaPID Assay System for PCB analysis can provide useful, cost-effective data.

In 2003, the USEPA used the RaPID Assay System at a Superfund site in South Dakota to more clearly establish whether the site posed potential risks because of previous evidence of PCB releases to soil, surface water, and sediment at the site. A clear correlation was found between the RaPID Assay System and contract laboratory program (CLP) results, with an r-value of 0.989. All field concentrations were found to be below action levels.

In 2002, the New Jersey Department of Environmental Protection (NJDEP) successfully used the RaPID Assay System at a Brownsfield site (NJDEP, 2005) to collect real time data to assist in the mapping of the boundaries of PCB "hot spots."

#### 5.6.3 Sediment Core Sampling

A sediment core will be collected in the eastern culvert using a piston core. The piston core will use a polycarbonate tube approximately 3 inches in diameter. The piston core will be advanced to a minimum of 3 feet or until refusal is met. The penetration of the sampling device and the length of the sediment core recovered will be recorded on the sediment core log form. Acceptance criteria for a sediment core sample are as follows:

- The core penetrated to its maximum length or to refusal.
- The amount of material retained was at least 50 percent of the length of core penetration.
- Cored material did not extend out the top of the core tube or contact any part of the sampling apparatus at the top of the core tube.
- There are no obstructions in the cored material that might have blocked the subsequent entry of sediment into the core tube and resulted in incomplete core collection.

The recovered core will be processed and samples will be collected on-site.

The core sample tubes will be decontaminated prior to use. The core tube caps will be removed immediately prior to placement into the coring device. Care will be taken during sampling to avoid contact of the sample tube with potentially contaminated surfaces. An extra sample tube will be available during sampling operations for uninterrupted sampling in the event of a core tube breakage or contamination. Core tubes suspected to have been accidentally contaminated will not be used. Core collection will be documented using forms and field notes. The following information will be included in the documentation:

- Mudline measurement taken at the sample location
- Location of each station as determined by measuring the distance from the upstream opening of the culvert to the core location

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- Date and time of collection of the sediment core sample
- Names of Field Supervisor and person(s) collecting and handling the sample
- Observations made during sample collection including weather conditions, complications, and other details associated with the sampling effort
- The sample station identification

- Penetration depth as noted by markings on the coring device
- Recovery for each core based on physical measurement of sediment in the recovered core
- Photographs of each recovered core
- Qualitative notation of apparent resistance of sediment column to coring
- Any deviation from the approved SAP

#### 5.6.3.1 Sediment Core Sample Processing

All working surfaces and instruments will be thoroughly cleaned, decontaminated, and covered with aluminum foil to minimize outside contamination between sampling events. Disposable gloves will be discarded after processing each station and replaced prior to handling decontaminated instruments or work surfaces.

Sample containers will be kept in packaging as received from the analytical laboratory until use; a sample container will be withdrawn only when a sample is to be collected and will be returned to a cooler containing completed samples.

The steps for processing the samples are listed below:

- 1. Cut core longitudinally using a power shears, taking care not to penetrate the sediment while cutting. Make two longitudinal cuts along the sides of the core so that the core can be opened to expose the sediment.
- 2. The sediment core will be split with a decontaminated stainless steel wire core splitter or decontaminated spatulas to expose the center of the two halves for sampling.
- 3. Record the description of the core sample on the core log form (Attachment 2) for the following parameters as appropriate and present:
  - Sample recovery (depth in feet of penetration and length of recovered sediment)
  - Sample geology
  - Odor (e.g., hydrogen sulfide, petroleum, etc.)
  - Vegetation
  - Debris

- Biological activity (e.g., detritus, shells, tubes, bioturbation, live or dead organisms)
- Presence of oil sheen
- Any other distinguishing characteristics or features
- 4. Divide core into 33-cm intervals so each interval can be sampled separately.
- 5. Using a clean stainless-steel spoon, place sample material from the core into a cleaned stainless-steel bowl, and homogenize by hand or by using a stainless steel paddle and variable speed drill for each 1-foot sample interval.
- 6. Using a clean, stainless-steel spoon, completely fill pre-labeled sample containers as indicated in Table 10 for the analyses.
- 7. Immediately after filling the sample container with sediment, place the screw cap on the sample container and tighten.
- 8. Thoroughly check all sample containers for proper identification, analysis type, and lid tightness.
- 9. Pack each container carefully to prevent breakage and place inside of a cooler with ice for storage at the proper temperature  $(4 \pm 2^{\circ}\text{C for all samples})$ .

# 5.6.4 Surface Water Sampling

Surface water collection will be conducted after sediment probing and sampling is complete. To minimize potential interferences from suspended particulate materials associated with the sediment probing, a 24-hour settling time between sediment sampling and water sampling will be implemented. In addition, samples will be collected during a slack or outgoing tide. Mid-depth samples will be collected at four locations. Because the water depths in these areas are generally very shallow (less than 2 feet) and previous water column sampling at the Site shows that the water column is generally well-mixed, it is assumed that mid-depth samples will adequately characterize the entire water column. Two of the four samples will be collected at the upstream and downstream ends of the area of interest, with two samples collected from locations identified by the field team based on a review of the field test results of the sediment sampling. A duplicate water sample will be collected from one location for quality assurance/quality control (QA/QC) purposes. The surface water samples collected during this task will be submitted to an analytical laboratory and analyzed for PCB

congeners, total organic carbon, and total suspended solids. Sampling locations will be determined and recorded using a DGPS.

Water samples will be collected using a horizontal van Dorn bottle or peristaltic pump (or similar type of surface water sampler). Collection of water using a van Dorn bottle and peristaltic pump are listed as potential methods in TCEQ 2003. The sampler will be lowered to the target water depth on a rope, allowed to sit at the target depth for a short period to allow any mudline particulates potentially disturbed during the deployment to settle out, and will be triggered close by the deployment of a messenger in the case of a van Dorn bottle, or the pump turned on for a peristaltic pump sampler.

Samples will be collected from mid-depth as determined in the field. The depths will be determined using a fathometer or lead line. If mid-depth is within 1 foot of the mudline, (i.e., water depth is less than 2 feet), water will be collected from a depth equal to one-third the water depth, per TCEQ guidance (TCEQ 2003) in lieu of the mid-depth samples. If excessive turbidity (relative to the natural turbidity of the water for that day) is observed in a collected sample, the sample will be discarded, and the sampler will be redeployed and allowed additional time for the disturbed bottom sediment to clear.

Water sample processing will occur directly after collection. The sampler may require a second deployment at each station if additional volume is needed to fill the required sample containers. The collected water will be poured into the appropriate pre-labeled sample containers, as outlined in Table 11.

#### 5.7 Decontamination

Decontamination procedures are outlined below for sediment and water sampling.

#### 5.7.1 Surface and Core Sediment Sampling

Sample collection equipment, containers, instruments, working surfaces, technician protective gear, and other items that may come into contact with sediment sample material must meet high standards of cleanliness. All equipment and instruments that are used in direct contact with the sediment collected for analysis will be made of glass, stainless steel,

high density polyethylene (HDPE), or polytetrafluoroethylene (PTFE), and will be cleaned prior to each day's use and between sampling or handling. Decontamination of all items will follow USEPA protocols (1986). The decontamination procedure is as follows:

- Pre-wash rinse with tap water or Site water
- Wash with solution of tap water and Liquinox soap (brush)
- Rinse with tap water
- Rinse three times with distilled water
- Cover (no contact) all decontaminated items with aluminum foil
- Stored in clean, closed container for next use

The Field Supervisor may elect to implement a hexane rinse if there are significant residues observed on field equipment after the above decontamination procedures are used.

#### 5.7.2 Surface Water Sampling

To prevent sample contamination, water sampling equipment will undergo the following decontamination procedures between each sampling station:

- Wash with phosphate-free detergent and tap water using a scrub brush
- Rinse with distilled water

# 5.8 Field Quality Assurance/Quality Control (QA/QC)

# 5.8.1 Field Quality Assurance Samples

Per the RI Work Plan QAPP (Anchor 2007a), field duplicates will be sampled and submitted for analysis at a frequency of five percent of samples submitted for each sample matrix (sediment and water). Temperature indicators will be included in each container for shipment of bulk sediment and water chemistry to the laboratory.

# 5.8.2 Performance Audits and Corrective Actions

Performance audits and corrective actions will be performed per the RI Work Plan QAPP (Section 16; Anchor 2007a).

#### 5.9 Investigation-Derived Waste Handling and Tracking

This section provides a waste management plan for handling investigation-derived waste associated with activities at the Site.

Investigation-derived waste for this SAP is expected to consist of:

- Excess sediment generated during sampling (grab samples and cores)
- Personal protective equipment (PPE) and other solid waste
- Decontamination and rinse water

#### 5.9.1 Sediment

Generation of some excess sample material is anticipated during collection of sediment grab samples and cores. Whenever possible, grab and core material will be returned to the environment by returning the sediment back to the collection site (Patrick Bayou).

Field sampling conditions may preclude safe disposal of excess material at the time of sampling. If needed, sediments and slurries will be retained and stored in lined 50-gallon drums, or other approved containers, for later disposal at an approved solid waste handling facility. Contents of the containers will be clearly marked. A log of collection dates and times, plus approximate volume of each sample, will be maintained to facilitate off-site disposal of the material as either non-hazardous or hazardous material.

#### 5.9.2 Decontamination and Rinse Water

Decontamination and rinse water will be retained in lined, 10-gallon buckets, and disposed of via the municipal sewer system if processing and decontamination occurs in the field. If processing and decontamination occurs in an on-site laboratory facility, decontamination and rinse water will be discharged directly into a sink tied to the municipal sewer system. Dilution of COPCs by flushing with tap water and the additional dilution of COPCs by other sources of flow in the municipal sewer system should be sufficient to achieve non-hazardous levels or no appreciable increase in ambient COPCs.

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Excess water from water sample collection will be returned to the Site.

#### 5.9.3 PPE and Other Solid Waste

PPE and other solid waste will be decontaminated to the extent possible and disposed of as municipal waste. If needed, PPE and solid waste with excess sediment or slurry contamination will be retained and stored in lined 10-gallon buckets. Retained PPE and solid waste will be stored for later disposal at an approved solid waste handling facility. Contents of the containers will be clearly marked. A log of collection dates and times, plus approximate volume of the waste, will be maintained to facilitate off-site disposal of the material as either non-hazardous or hazardous material.

#### **6 MEASUREMENT AND DATA ACQUISITION**

This section describes the procedures for field documentation, sample identification, and handling/transporting sediment and surface water samples to the analytical laboratory.

#### 6.1 Field Documentation and Sample Identification

Field sample logs and notebooks will be maintained for all samples collected during the field program. All sample field notebooks will have numbered pages. All data entries will be made using indelible-ink pens. Corrections will be made by drawing a single line through the error, writing in the correct information, then dating and initialing the change.

#### 6.1.1 Sediment Sample Documentation

At a minimum, the following information will be included in the log for each sediment grab or core:

- 1. The sample station number
- Location of each sample station as determined by DGPS (with proper description of measurement units)
- 3. Date and collection time of each sediment sample
- 4. Names of Field Supervisor and person(s) collecting and logging sample
- 5. Observations made during sample collection including weather conditions, complications, and other details associated with the sampling effort
- 6. Recovery of sediment grab and length and recovery for the sediment core
- Qualitative notation of apparent resistance of sediment column to coring/sampling, including notes on debris
- 8. Any deviations from the approved SAP

During sediment sample processing, the following information should be recorded in the sample log sheet or field log:

- 1. Sample recovery
- 2. Physical soil description in accordance with the Unified Soil Classification System (includes soil type, density/consistency of soil, and color)
- 3. Odor (e.g., hydrogen sulfide, petroleum, etc.) if any is passively observed

- 4. Vegetation
- 5. Debris
- 6. Biological activity (e.g., detritus, shells, tubes, bioturbation, or live or dead organisms)
- 7. Presence and depth of the redox potential discontinuity layer
- 8. Presence of oil sheen
- Any other distinguishing characteristics or features with photographs taken to support field descriptions

# 6.1.2 Sediment Probing Sample Identification

The identification scheme for sediment probing locations will contain "PBUC," to depict this investigation (Patrick Bayou Upstream Characterization), followed by sequential numbers assigned in the field. For example, PBUC001 will be the first sediment probing location and PBUC020 will be the twentieth sediment probing location. The probing will begin at the most downstream portion of the area of interest (PBUC066), so probing station identifiers will increase sequentially from downstream to upstream.

# 6.1.3 Sediment Sample Identification

Sample identification will include the sediment probing sample name, depth interval information and sample date. For example, for sample PBUC###-#XX###-YYMMDD-X:

- PBUC###-#XX###-YYMMDD-X: Each location will be identified by PBUC, to depict this investigation (Patrick Bayou Upstream Characterization), followed by the number assigned to the sediment probing location (e.g., PBUC012).
- PBUC###-#XX###-YYMMDD-X: Individual samples at each location will be identified by the same alphanumeric identifier used to identify the stations, followed by a one-digit numeric substation identifier, a two-digit matrix identifier (i.e., SS = surface sediment grab, SC = sediment core), and a three-digit number identifying the lower interval measurement (in cm) for that sample.
- PBUC###-#XX###-YYMMDD-X: The collection date of each sample will be included in the sample name to distinguish between previously collected samples from the same station. An alphanumeric identifier will follow the date, indicating the sample type:

- N = Normal sample
- D = Field duplicate or homogenization split of the normal sample

This information is included in detail for planned samples in Table 2.

Upon return from the field, the sediment sample locations will be plotted using a Geographical Information Systems (GIS) software program. This distance from each sediment sample location to the mouth of Patrick Bayou will be measured and used to assign each sediment station with a station identifier using project-specific nomenclature (PBUC to depict project location, Patrick Bayou, and the channel station in hundreds of feet), for example, PBUC075.

# 6.1.4 Surface Water Sample Documentation

At a minimum, the following information will be included in the log for surface water grabs (Attachment 2):

- 1. The sample station number
- 2. Location of each sample station as determined by DGPS (with proper description of measurement units)
- 3. Date and collection time of each water sample
- 4. Names of Field Supervisor and person(s) collecting and logging the sample
- 5. Observations made during sample collection including: weather conditions, bayou conditions, complications, and other details associated with the sampling effort, such as descriptions of any upstream outfalls operating during sampling and conditions of the receiving waters near the outfall
- 6. Water depth and sample depth
- 7. Position in tidal event (e.g., time before or after low tide)
- 8. Any deviations from the approved SAP

# 6.1.5 Surface Water Sample Identification

Sample identification for surface water samples will include depth interval information and will follow a scheme similar to the one for sediment samples as described below. For example, for sample PB##-#XX###-YYMMDD-X:

- PB###-#XX###-YYMMDD-X: Each location will be identified by PB, to depict the project location (Patrick Bayou), and the station identifier associated with the channel station in hundreds of feet (e.g., PB100).
- PB###-#XX###-YYMMDD-X: Individual samples at each location will be identified by the same alphanumeric identifier used to identify the stations, followed by a one-digit numeric substation identifier, a two-digit matrix identifier (i.e., SW = surface water grab), and a three-digit number identifying the position in the water column (i.e., MID = mid depth of the water column) for that sample.
- PB##-#XX###-YYMMDD-X: The collection date of each sample will be included in the sample name to distinguish between previously collected samples from the same station. An alphanumeric identifier will follow the date, indicating the sample type:
  - N = Normal sample
  - D = Field duplicate or homogenization split of the normal sample

This information is included in detail for planned samples in Table 3.

# 6.2 Sample Handling and Transport

As described in the RI Work Plan QAPP (Anchor 2007a), components of sample custody procedures include the use of field log books, sample labels, custody seals, and chain-of-custody (COC) forms. Each person involved with sample handling will be trained in COC procedures before the start of the field program. The COC form will accompany the samples during shipment from the field to the laboratory.

# 6.2.1 Field Custody

The following procedures will be used to document, establish, and maintain custody of field samples:

- 1. Sample labels will be completed for each sample with waterproof ink, making sure that the labels are legible and affixed firmly on the sample container.
- 2. All sample-related information will be recorded in the project log book.
- 3. The field sampler will retain custody of the samples until they are transferred or properly dispatched.

- 4. To simplify the COC record and minimize potential problems, as few people as possible should handle the samples. For this reason, one individual from the field sampling team will be designated as the responsible individual for all sample transfer activities. This field investigator will be responsible for the care and custody of the samples until they are properly transferred to another person or facility.
- 5. A COC form will accompany all samples. This record documents the transfer of custody of samples from the field sampler to the laboratory. When transferring the possession of samples, the individuals relinquishing the receiving will sign, date, and note the time on the record.
- 6. Samples will be properly packaged for shipment and sent to the appropriate laboratory for analysis with a separate signed COC form, enclosed in a plastic bag, and taped inside the cover of each sample box or cooler. The original record will accompany the shipment, and a copy will be retained by the Field Supervisor. When samples are relinquished to shipping companies for transport, the tracking number will be recorded on the COC form.
- 7. The COC must be signed when relinquished by field personnel and signed by the laboratory receiving the samples.
- 8. Custody seals will be used on the shipping containers when samples are shipped to the laboratory to inhibit sample tampering during transportation.

# 6.2.2 Laboratory Sample Custody

Each laboratory receiving samples for this project must comply with the laboratory sample custody requirements outlined in its Quality Assurance Plan (QAP). The laboratory will designate a sample custodian who will be responsible for maintaining custody of the samples and all associated records documenting that custody. In addition, the laboratory will provide the following quality checks:

- Each laboratory will check to see that there has been no tampering with the custody seals on the coolers.
- Upon receipt of the samples, the custodian will check the original COC and requestfor-analysis documents and compare them with the labeled contents of each sample container for corrections and traceability. The sample custodian will sign the COC and record the date and time received in the 'Received by Laboratory' box.

- The sample custodian will assign a unique laboratory sample number to each sample.
- Cooler temperature will be checked and recorded.
- Care will be exercised to annotate any labeling or descriptive errors. If discrepancies
  occur in the documentation, the laboratory will immediately contact the sample
  tracking coordinator and Anchor QEA Quality Assurance Manager as part of the
  corrective action process. A qualitative assessment of each sample container will be
  performed to note anomalies, such as broken or leaking bottles. This assessment will
  be recorded as part of the incoming COC procedure.

Samples will be stored in a secured area and at a temperature of  $4^{\circ} \pm 2^{\circ}$ C, if necessary, until analyses are to begin. Unless otherwise specified by the Project Manager, samples will be retained for a period of 60 days after the final report is released by the laboratory, after which they will be disposed in accordance with the laboratory Standard Operating Procedures (SOP) for waste disposal. Samples submitted to the laboratory marked for archive on the COC and sample container will be frozen and stored until further notice.

# 6.2.3 Sample Packaging and Shipping

During the field efforts, the Anchor QEA Quality Assurance Manager will notify the appropriate laboratories about sample shipments.

Hard plastic ice chests or coolers with similar durability will be used for shipping samples. The coolers must be able to withstand a 4-foot drop onto solid concrete in the position most likely to cause damage. Samples will be double-bagged in Ziploc bags and grouped by sample set. Styrofoam or bubble wrap will be used as packaging material to protect the samples from leakage during shipment. A volume of ice approximately equal to the sample volume should be present in each cooler. Blue ice will not be used. After packing is complete, the cooler will be taped securely, with custody seals affixed across the top and bottom joints. In addition, these procedures will be followed when packing coolers of sample for shipping:

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- 1. Include absorbent material in the cooler to absorb any ice melt.
- 2. Record the airbill on each COC.
- 3. List the appropriate contact person on the COC.
- 4. Use custody seals on the cooler.

Samples will be shipped priority overnight Federal Express or transported by courier (or equivalent) to the laboratory.

#### 7 REFERENCES

- Anchor Environmental, LLC (Anchor), 2006. Preliminary Site Characterization Report.

  Patrick Bayou Superfund Site. Deer Park, Texas. Prepared for USEPA and Patrick Bayou Joint Defense Group.
- Anchor, 2007a. Remedial Investigation Work Plan. Patrick Bayou Superfund Site. Deer Park, Texas. Prepared for USEPA and Patrick Bayou Joint Defense Group. January 8, 2007.
- Anchor, 2007b. Work Package 2 Vertical Profiling Hydrodynamic Field Data Collection and Contaminant Source Evaluation Data Report. Patrick Bayou Superfund Site, Deer Park, Texas. Prepared for USEPA and Patrick Bayou Joint Defense Group. April 5, 2007.
- Anchor, 2008a. Draft Selection of Chemicals of Potential Concern for Ecological Risk Assessment. Patrick Bayou Superfund Site, Deer Park, Texas. Prepared for USEPA and Patrick Bayou Joint Defense Group. April 3, 2008.
- Anchor, 2008b. Draft Mixing Zone Evaluation Work Plan. Patrick Bayou Superfund Site, Deer Park, Texas. Prepared for U.S. Environmental Protection Agency and Patrick Bayou Joint Defense Group. October 2008.
- Anchor QEA, LLC, 2009a. Patrick Bayou Sediment Mixing Zone Layer Study. Memorandum to U.S. Environmental Protection Agency. June 15, 2009.
- Anchor QEA, 2009b. Draft Patrick Bayou Sediment Transport Modeling Report. Prepared for U.S. Environmental Protection Agency and the Patrick Bayou Joint Defense Group. April 2009.
- Anchor QEA, 2009c. Health and Safety Plan. Patrick Bayou Superfund Site, Deer Park, Texas. Prepared for U.S. Environmental Protection Agency and the Patrick Bayou Joint Defense Group. October 2009.
- Anchor QEA, 2010. Sediment and Surface Water Contaminant of Potential Concern Delineation Data Report. Patrick Bayou Superfund Site, Deer Park, Texas. Prepared for Patrick Bayou Joint Defense Group. May 2010.

- New Jersey Department of Environmental Protection (NJDEP), 2005. Characterization of Volatile Organic Compounds (VOC) and Polychlorinated Biphenyls (PCB) Using Immunoassay PCB Test Kits and Field Gas Chromatography (GC) Analysis at the Albert Steel Drum Site, Newark, New Jersey.
- Texas Commission on Environmental Quality (TCEQ), 2003. Surface Water Quality Monitoring Procedures, Volume I: Physical and Chemical Monitoring Methods. RG-415. Water Quality Planning Division.
- U.S. Environmental Protection Agency (USEPA), 1986. General QA/QC consideration for collecting environmental samples in Puget Sound. U.S. Environmental Protection Agency, Region 10, Office of Puget Sound Estuary Program, Seattle, WA.
- U.S. Environmental Protection Agency (USEPA), 1997. Recommended Quality Assurance and Quality Control Guidelines for the Collection of Environmental Data in Puget Sound. In: Recommended protocols for measuring selected environmental variables in Puget Sound. U.S. Environmental Protection Agency, Puget Sound Estuary Program, Seattle, WA.
- U.S. Environmental Protection Agency (USEPA), 1998. Environmental Technology Verification Report. Immunoassay Kit, Strategic Diagnostics, Inc. RaPID Assay System for PCB Analysis. EPA/600/R-98. August.

# **TABLES**

Table 1
Project Personnel Quality Assurance Responsibilities

Title	Responsibility	Name/ Affiliation	Contact Information
Project Coordinator	Responsible for coordination of schedule, budget, and facilitating technical discussions between agencies, Anchor QEA, and Patrick Bayou Joint Defense Group.	Bob Piniewski	Project Navigator 70 Traylee Wake Forest, NC 27587 bobp@projectnavigator.com
Anchor QEA Project Director	Responsible for the overall delivery of project objectives in alignment with the operating parameters set forth in the project QAPP.	David Keith	Anchor QEA, LLC 614 Magnolia Avenue Ocean Springs, MS 39564 (228) 818-9626 dkeith@anchorqea.com
Anchor QEA Project Manager	Responsible for the coordination and execution of all work items associated with project planning and implementation. Liaison between program-level managers and project-level team members. Identifies team members and project assignments. Manages and tracks schedule and budget. Ensures that all tasks are completed by assigned team members within schedule and budget constraints.	David Keith	Anchor QEA, LLC 614 Magnolia Avenue Ocean Springs, MS 39564 (228) 818-9626 dkeith@anchorqea.com
Anchor QEA Project Health and Safety Manager	Responsible for overseeing health and safety program for field tasks associated with RI/FS. Reviews Site Health and Safety Plan, Site job safety analyses and training requirements.	Chris Torell	Anchor QEA, LLC 290 Elwood Davis Road Suite 340 Liverpool, NY 13088 (315) 453-9009 ctorell@anchorqea.com
Anchor QEA Project QA Manager	Responsible for Data Quality Objective planning, QAPP development, ensuring the project objectives are met. Liaison between project manager and project team. Task lead for data interpretation and final report preparation.	Delaney Peterson	Anchor QEA, LLC 720 Olive Way, Suite 1900 Seattle, WA 98101 (206) 287-9130 dpeterson@anchorgea.com
Anchor QEA Data Manager	Point of contact for all issues concerning database maintenance, data loading, verifying data, and communicating with project team regarding database and data content issues.	Lisa Allen	Anchor QEA, LLC 720 Olive Way, Suite 1900 Seattle, WA 98101 (206) 287-9130 lallen@anchorgea.com
Field Supervisor	Responsible for field data collection. In addition, the Field Supervisor will implement the Health and Safety Plan in the field.	Jason Kase	Anchor QEA, LLC 4208 Cherry Laurel Drive Pensacola, FL 32054 (850) 912-8400 ikase@anchorgea.com
Project Emergency Coordinator	Responsible for managing potential emergency situations during field work for the RI/FS. Includes notifying appropriate Points of Contact at each facility and the Project Manager in case of fire, spills, personal injury, or any other emergency situation that may arise.	Jason Kase	Anchor QEA, LLC 4208 Cherry Laurel Drive Pensacola, FL 32054 (850) 912-8400 jkase@anchorqea.com
Vessel Operator	Responsible for the safe operation of boats or other sampling platforms utilized during sampling and maintenance activities. Will ensure that proper safety equipment is on the vessel and operating correctly and that all personnel on the boat are familiar with safety procedures, features, and equipment.	Jason Kase	Anchor QEA, LLC 4208 Cherry Laurel Drive Pensacola, FL 32504 (850) 912-8400 jkase@anchorqea.com

Table 2
Summary of Sediment Sampling Study Design

		Station Co	ordinates <sup>a</sup>	λ			>-				S		s <sub>d</sub>
Sample ID <sup>b</sup>	Depth Interval <sup>c</sup> (cm)	Northing	Easting	PCB Field Assay	Grain Size	тос	Specific Gravity	Mercury	Total Metals <sup>d</sup>	PAHs <sup>d</sup>	PCB Congeners	PCB Aroclors <sup>d</sup>	Dioxins/Furans <sup>d</sup>
Patrick Bayou Channel Sediment Samples <sup>e</sup>													
PBUC###-1SS010-N	0-10	TBD	TBD	Χ						Χ		Χ	
Culvert Sediment Samp	les												
PBUC###-1SS010-N	0-10	TBD	TBD		Χ	Χ	Χ	Χ	Χ	Χ	Χ		Х
PBUC###-1SS010-N	0-10	TBD	TBD		Χ	Χ	Χ	Χ	Χ	Χ	Χ		Χ
PBUC###-1SS010-N	0-10	TBD	TBD		Х	Χ	Х	Х	Х	Χ	Χ		Χ
PBUC###-1SS010-N	0-10	TBD	TBD		Χ	Χ	Χ	Χ	Χ	Х	Χ		Χ
PBUC###-1SC###-N	0-30	TBD	TBD		Χ	Χ	Χ	Χ	Χ	Χ	Χ		Χ
PBUC###-1SC###-N	30-60	TBD	TBD		Χ	Χ	Χ	Χ	Χ	Х	Χ		Χ
PBUC###-1SC###-N	60-90	TBD	TBD		Χ	Χ	Χ	Χ	Χ	Χ	Χ		Χ
Field Quality Assurance	Field Quality Assurance/Quality Control Samples												
PBUC###-1SS010-D <sup>f</sup>	0-10	TBD	TBD	Χ						Χ		Χ	

- a Station Coordinates will be State Plane coordinates based on North American Datum (NAD) 83 for Texas, South Central.
- b Sequential numbers based on sediment probing locations will be assigned to sample IDs in the field. Project specific nomenclature will be assigned upon return from the field. See Section 6.1.3.
- c Specific station and interval may be changed in the field to best represent site conditions.
- d See Table 4 for complete list of analytes included in analyses
- e Up to 36 sediment grab samples will be collected and analyzed for total PCB congeners in the field using the PCB Field Assay Test. Of these, six will be selected for analysis by an off-site laboratory for select PAHs and PCB Aroclors.
- f Location to be determined in the field based on Site conditions. Samples are to be named in accordance with Section 6.1.3

Table 3
Summary of Surface Water Sampling Study Design

		Station Co	S					
Station ID	Sample ID	Northing	Easting	PCB Congeners	T0C	TSS		
PB066	PB066-1SWMID-N	13831288.66	3201385.637	Х	Χ	Χ		
PB### <sup>b</sup>	PB###-1SWMID-N	TBD	TBD	Х	Х	Х		
PB### <sup>b</sup>	PB###-1SWMID-N	TBD	TBD	Х	Х	Χ		
PB101	PB101-1SWMID-N	13828184	3201303.387	Х	Х	Х		
Field Quality Assurance/Quality Control Samples								
PB### <sup>b</sup>	PB###-1SWMID-D	TBD	TBD	Х	Χ	Х		

- a Station coordinates are State Plane coordinates based on North American Datum (NAD) 83 for Texas, South Central
- b Location to be determined in the field based on Site conditions. Samples are to be named in accordance with Section 6.1.5

**TOC - Total Organic Carbon** 

TSS - Total Suspended Solids

TBD - To Be Determined

Table 4
Parameters for Analysis and Target Practical Quantitation Limits for Sediment

Parameter	Units <sup>a</sup>	Sediment Target PQL	Analytical Method
Conventional Parameters			
Grain Size	%	0.1	ASTM D422
Total Organic Carbon	%	0.02	9060/415.1
Specific Gravity			ASTM D854
Metals			
Mercury	mg/kg	0.05	7471A
Arsenic	mg/kg	0.2	6010B/6020
Cadmium	mg/kg	0.2	6010B/6020
Chromium	mg/kg	0.5	6010B/6020
Copper	mg/kg	0.5	6010B/6020
Lead	mg/kg	1.0	6010B/6020
Nickel	mg/kg	0.5	6010B/6020
Selenium	mg/kg	0.5	6010B/6020
Zinc	mg/kg	4.0	6010B/6020
Polycyclic Aromatic Hydrocarbons			
LPAH			
2-Methylnaphthalene	μg/kg	6.7	8270C/ SIM
Acenaphthene	μg/kg	6.7	8270C/ SIM
Acenaphthylene	μg/kg	6.7	8270C/ SIM
Anthracene	μg/kg	6.7	8270C/ SIM
Fluorene	μg/kg	6.7	8270C/ SIM
Naphthalene	μg/kg	6.7	8270C/ SIM
Phenanthrene	μg/kg	6.7	8270C/ SIM
НРАН	-	•	
Benzo(a)anthracene	μg/kg	6.7	8270C/ SIM
Benzo(a)pyrene	μg/kg	6.7	8270C/ SIM
Benzo(b)fluoranthene	μg/kg	6.7	8270C/ SIM
Benzo(e)pyrene	μg/kg	6.7	8270C/ SIM
Benzo(g,h,i)perylene	μg/kg	6.7	8270C/ SIM
Benzo(k)fluoranthene	μg/kg	6.7	8270C/ SIM
Chrysene	μg/kg	6.7	8270C/ SIM
Dibenzo(a,h)anthracene	μg/kg	6.7	8270C/ SIM
Indeno(1,2,3-cd)pyrene	μg/kg	6.7	8270C/ SIM
Fluoranthene	μg/kg	6.7	8270C/ SIM
Perylene	μg/kg	6.7	8270C/ SIM
Pyrene	μg/kg	6.7	8270C/ SIM
Alkyl-substituted PAH homologs			
C1-Chrysenes	μg/kg	63	8270C/ SIM
C1-Fluoranthene/Pyrene	μg/kg	63	8270C/ SIM
C1-Fluorenes	μg/kg	63	8270C/ SIM
C1-Naphthalenes	μg/kg	63	8270C/ SIM
C1-Phenanthrenes/Anthracenes	μg/kg	63	8270C/ SIM
C2-Chrysenes	μg/kg	63	8270C/ SIM

Table 4
Parameters for Analysis and Target Practical Quantitation Limits for Sediment

Parameter	Units <sup>a</sup>	Sediment Target PQL	Analytical Method
C2-Fluorenes	μg/kg	63	8270C/ SIM
C2-Naphthalenes	μg/kg	63	8270C/ SIM
C2-Phenanthrenes/Anthracenes	μg/kg	63	8270C/ SIM
C3-Chrysenes	μg/kg	63	8270C/ SIM
C3-Fluorenes	μg/kg	63	8270C/ SIM
C3-Naphthalenes	μg/kg	63	8270C/ SIM
C3-Phenanthrenes/Anthracenes	μg/kg	63	8270C/ SIM
C4-Chrysenes	μg/kg	63	8270C/ SIM
C4-Naphthalenes	μg/kg	63	8270C/ SIM
C4-Phenanthrenes/Anthracenes	μg/kg	63	8270C/ SIM
PCBs	-		
PCB Congeners 1-209	pg/g	10	1668
Aroclor 1016	μg/kg	4	8082
Aroclor 1221	μg/kg	4	8082
Aroclor 1232	μg/kg	4	8082
Aroclor 1242	μg/kg	4	8082
Aroclor 1248	μg/kg	4	8082
Aroclor 1254	μg/kg	4	8082
Aroclor 1260	μg/kg	4	8082
Dioxins and Furans	-		
1,2,3,4,6,7,8-HpCDD	pg/g	2.5	1613B
1,2,3,4,6,7,8-HpCDF	pg/g	2.5	1613B
1,2,3,4,7,8,9-HpCDF	pg/g	2.5	1613B
1,2,3,4,7,8-HxCDD	pg/g	2.5	1613B
1,2,3,4,7,8-HxCDF	pg/g	2.5	1613B
1,2,3,6,7,8-HxCDD	pg/g	2.5	1613B
1,2,3,6,7,8-HxCDF	pg/g	2.5	1613B
1,2,3,7,8,9-HxCDD	pg/g	2.5	1613B
1,2,3,7,8,9-HxCDF	pg/g	2.5	1613B
1,2,3,7,8-PeCDD	pg/g	2.5	1613B
1,2,3,7,8-PeCDF	pg/g	2.5	1613B
2,3,4,6,7,8-HxCDF	pg/g	2.5	1613B
2,3,4,7,8-PeCDF	pg/g	2.5	1613B
2,3,7,8-TCDD	pg/g	0.5	1613B
2,3,7,8-TCDF	pg/g	0.5	1613B
OCDD	pg/g	5.0	1613B
OCDF	pg/g	5.0	1613B

a - All chemical concentrations to be determined on a dry weight basis.

mg/kg - milligrams per kilogram

μg/kg - micrograms per kilogram

pg/g - picogram per gram

Table 5
Parameters for Analysis and Target Practical Quantitation Limits for Surface Water

Parameter	Units	<b>Detection Limit</b>	Analytical Method
Conventional Parameters			
Total Organic Carbon	mg/L	5.0	9060/415.1
Total Suspended Solids	mg/L	1.0	160.2
PCBs			
PCB Congeners 1-209	pg/L	10	1668

mg/kg - milligrams per kilogram pg/g - picogram per gram

Table 6
Laboratory Quality Control Sample Summary for Sediment

Analysis Type	Initial Calibration	Ongoing Calibration	Replicates	Matrix Spikes	SRM/LCS	Matrix Spike Duplicates	Method Blanks	Surrogate Spikes
Grain Size	Each batch <sup>a</sup>	NA	1 per 20 samples	NA	NA	NA	NA	NA
Specific Gravity	Each batch <sup>a</sup>	NA	1 per 20 samples	NA	NA	NA	NA	NA
Total Organic Carbon	Daily or each batch	1 per 10 samples	1 per 20 samples	1 per 20 samples	1 per 20 samples	NA	1 per 20 samples	NA
Metals	Daily	1 per 10 samples	1 per 20 samples	1 per 20 samples	1 per 20 samples	NA	1 per 20 samples	NA
Mercury	Daily	1 per 10 samples	1 per 20 samples	1 per 20 samples	1 per 20 samples	NA	1 per 20 samples	NA
Dioxin/Furans	As needed <sup>b</sup>	Every 12 hours	NA	NA <sup>c</sup>	NA <sup>c</sup>	NA <sup>c</sup>	1 per 20 samples	NA <sup>c</sup>
PCB Congeners	As needed <sup>b</sup>	Every 12 hours	NA	NA <sup>c</sup>	NA <sup>c</sup>	NA <sup>c</sup>	1 per 20 samples	NA <sup>c</sup>
PCB Aroclors	As needed <sup>b</sup>	1 per 10 samples	NA	1 per 20 samples	1 per 20 samples	1 per 20 samples	1 per 20 samples	Every sample
Semivolatile Organics	As needed <sup>b</sup>	Every 12 hours	NA	1 per 20 samples	1 per 20 samples	1 per 20 samples	1 per 20 samples	Every sample

- a Calibration and certification of drying ovens and weighing scales are conducted bi-annually.
- b Initial calibrations are considered valid until the ongoing continuing calibration no longer meets method specifications. At that point, a new initial calibration is performed.
- c Isotope dilution required per method
- NA Not Applicable
- SRM Standard reference material
- LCS Laboratory control sample

Table 7
Laboratory Quality Control Sample Summary for Surface Water

		Ongoing			_	Matrix Spike		Surrogate	
Analysis Type	Initial Calibration	Calibration	Replicates	Matrix Spikes	SRM/LCS	Duplicates	Method Blanks	Spikes	
Total Suspended Solids		NA	1 per 20	NA	NA	NA	NA	NA	
Total Suspended Solids	Each batch <sup>a</sup>	NA	samples		IVA	IVA	IVA	IVA	
Total Organic Carbon	Daily or each batch	1 per 10	1 per 20	1 per 20	1 per 20	NA	1 per 20	NA	
Total Organic Carbon	Daily of each batch	samples	samples	samples	samples	INA	samples		
DCD Congonors	A a mandad b	Every 12	NIA C	NIA C	NI A C	1 per 20	A. A. C		
PCB Congeners	As needed <sup>b</sup>	hours	NA N	NA <sup>c</sup>	NA	NA <sup>c</sup>	samples	NA <sup>c</sup>	

- a Calibration and certification of drying ovens and weighing scales are conducted bi-annually.
- b Initial calibrations are considered valid until the ongoing continuing calibration no longer meets method specifications. At that point, a new initial calibration is performed.
- c Isotope dilution required per method
- NA Not Applicable
- SRM Standard reference material
- LCS Laboratory control sample

Table 8

Data Quality Objectives for Sediment Samples

Parameter	Precision	Accuracy	Completeness
Grain Size	±20% RPD	NA	95%
Specific Gravity	±20% RPD	NA	95%
Total Organic Carbon	±20% RPD	75-125% R	95%
Metals	± 30% RPD	75-125% R	95%
Mercury	± 30% RPD	75-125% R	95%
Dioxin/Furans	± 35% RPD	50-150% R	95%
PCB Congeners	± 35% RPD	50-150% R	95%
PCB Aroclors	± 35% RPD	50-150% R	95%
Semivolatile Organics	± 35% RPD	50-150% R	95%
Alkylated PAHs	± 35% RPD	50-150% R	95%

RPD - Relative percent difference

R - Recovery

Table 9

Data Quality Objectives for Surface Water Samples

Parameter	Precision	Accuracy	Completeness
Total Suspended Solids	±20% RPD	NA	95%
Total Organic Carbon	±20% RPD	80-120% R	95%
PCB Congeners	± 30% RPD	60-140% R	95%

RPD - Relative percent difference

R - Recovery

Table 10
Sediment Sample Containers, Preservatives, and Holding Times

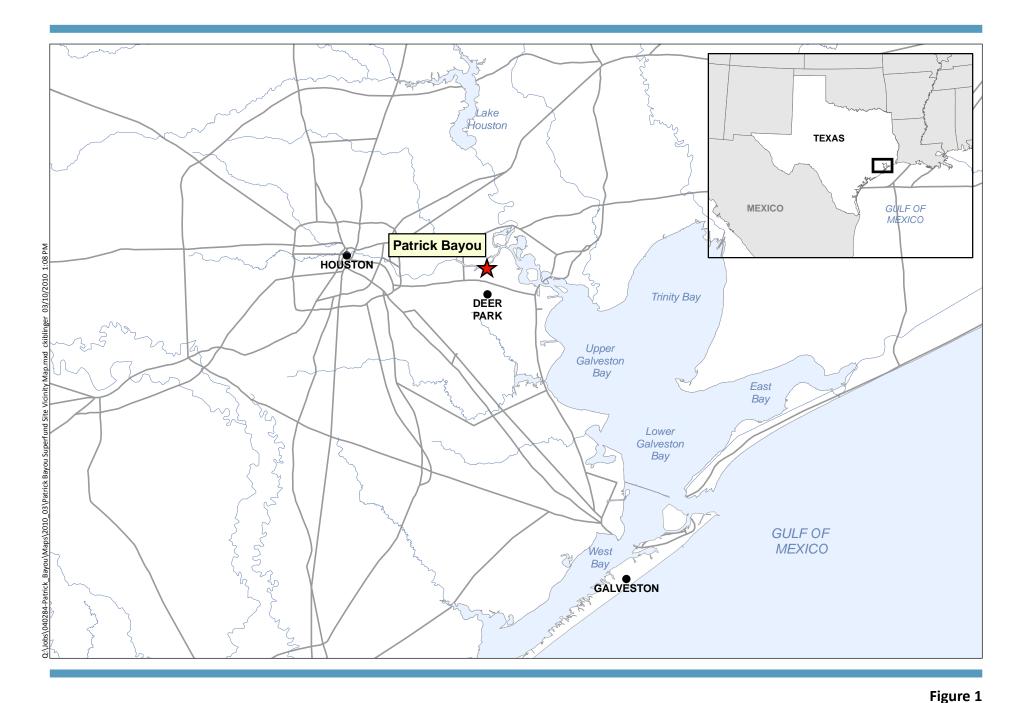
Parameter	Sample Size	Container Size and Type	Holding Time	Sample Preservation Technique
Total metals (with Hg)	50 g	4-oz Glass	6 months; 28 days for Hg	Cool/4° C
Total metals (with rig)	30 g	4-02 Glass	2 years (except Hg)	Freeze -18°C
Comivolatile organic			14 days until extraction	Cool/4° C
Semivolatile organic compounds (SVOC)	150 g	16-oz Glass	1 year until extraction	Freeze -18°C
compounds (3voc)			40 days after extraction	Cool/4° C
		f 6\/06	14 days until extraction	Cool/4° C
Alkylated PAHs	150 g	from SVOC container	1 year until extraction	Freeze -18°C
		Container	40 days after extraction	Cool/4° C
			14 days until extraction	Cool/4° C
PCB Aroclors	150 g	16-oz Glass	1 year until extraction	Freeze -18°C
			40 days after extraction	Cool/4° C
Total organic carbon	50 g	from GS	14 days	Cool/4° C
Total organic carbon	30 g	container	6 months	Freeze -18°C
Dioxins/Furans	150 g	8-oz Glass	1 year to extraction	Freeze -10°C
Dioxilis/Fulalis	130 g	6-02 Glass	1 year after extraction	Freeze -10°C
PCB Congeners	150 g	From dioxins	1 year to extraction	Freeze -10°C
container		container	1 year after extraction	Freeze -10°C
Specific gravity	100 g	From GS	6 months	
Grain size (GS)	300 g	16-oz HDPE		

-- Not Applicable

Table 11
Surface Water Sample Containers, Preservatives, and Holding Times

Parameter	Sample Size	Container Size and Type	Holding Time	Sample Preservation Technique
Total suspended solids	1L	1L HDPE	7 days	Cool/4° C
Total organic carbon	100 mL	250 mL Amber glass	28 days	Cool/4° C; H <sub>2</sub> SO <sub>4</sub> to pH<2
PCB Congeners	1L	1L Amber glass	1 year to extraction	Cool/4° C
r CD Congeners	IL	TE Allibei glass	1 year after extraction	Freeze -10°C

# **FIGURES**



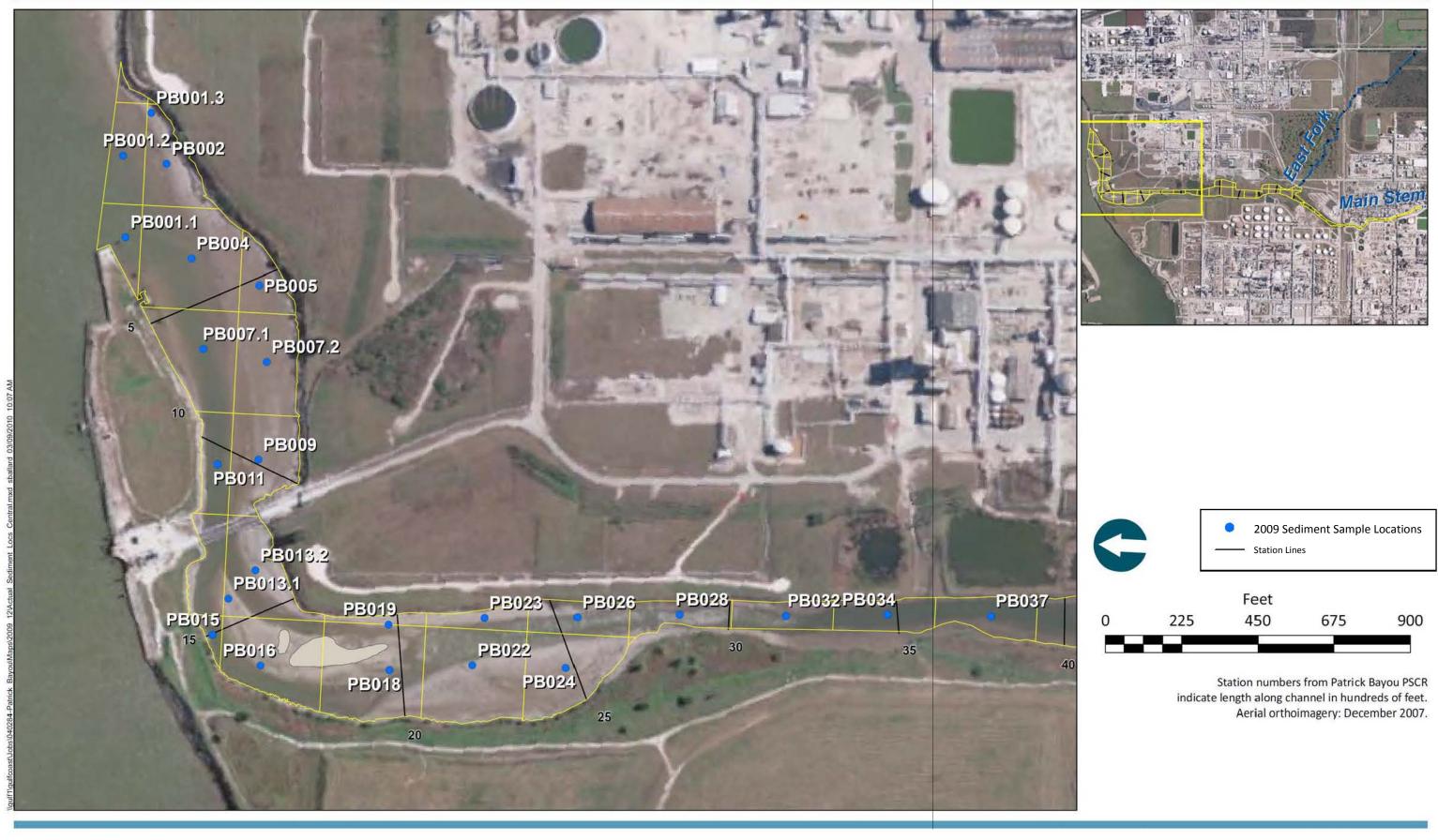




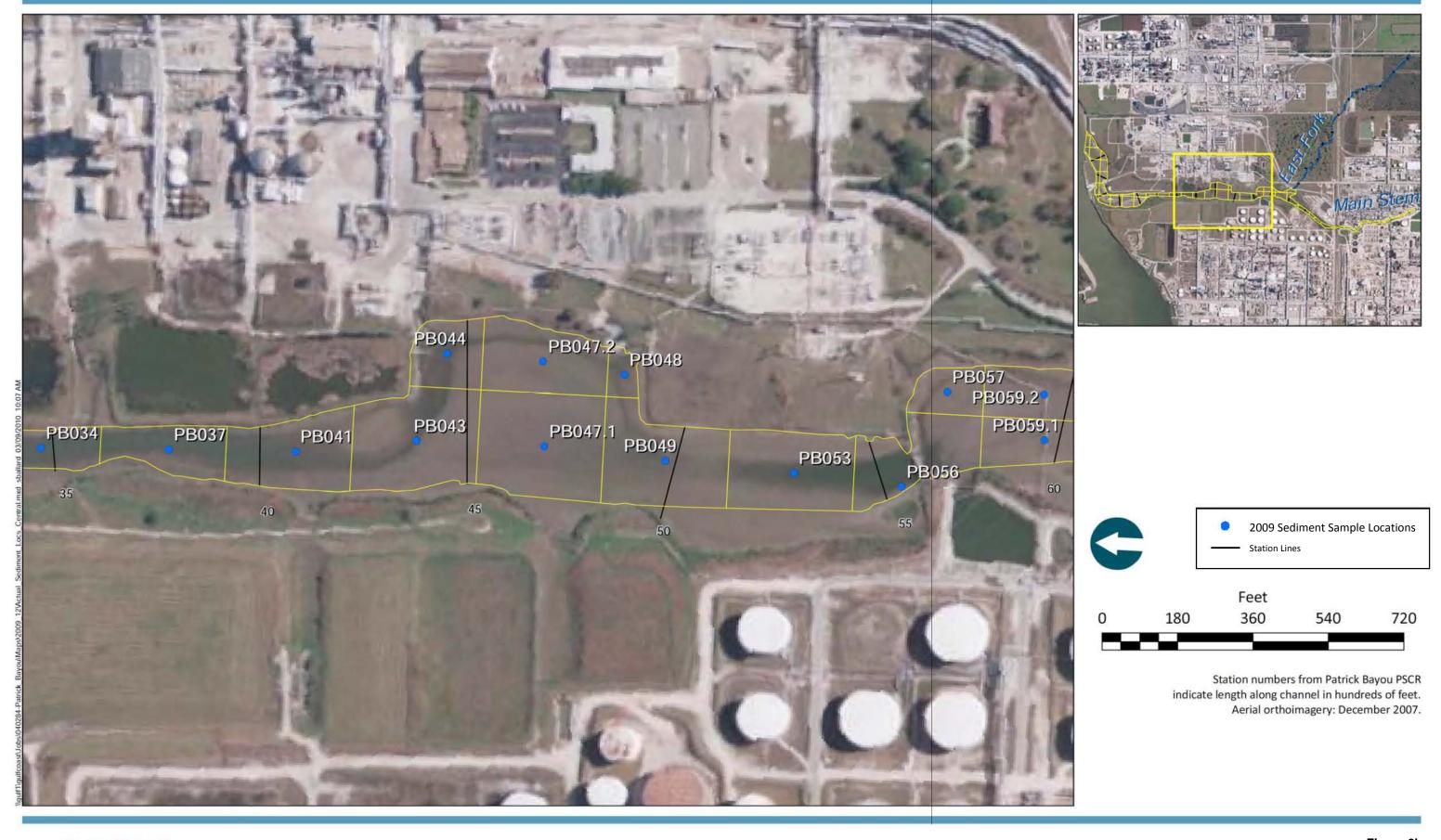


Patrick Bayou Superfund Site Vicinity Map

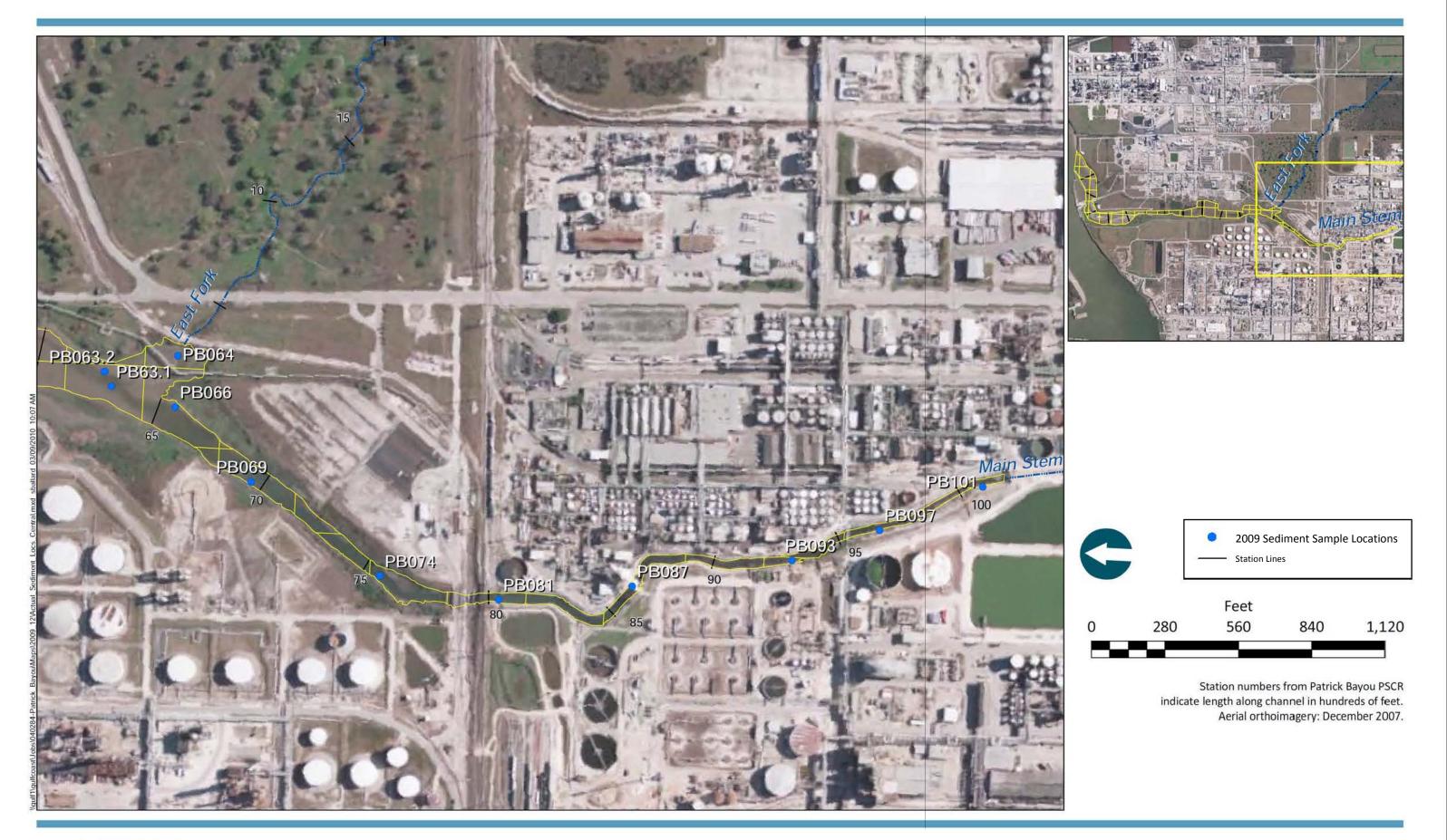
Upstream Patrick Bayou Characterization Sampling and Analysis Plan Patrick Bayou Superfund Site, Deer Park, Texas





















### NOTES:

1. Two additional surface water sample locations will be added based on review of sediment field test results. 2. Aerial Imagery: ESRI/Aerials Express, 2008.



Sediment Probing and Surface Water Sample Locations Upstream Patrick Bayou Characterization Sampling and Analysis Plan Patrick Bayou Superfund Site, Deer Park, Texas

# ATTACHMENT 1 ADDENDUM TO SITE HEALTH AND SAFETY PLAN



290 Elwood Davis Road, Suite 340 Liverpool, New York 13088 Phone 315.453.9009 Fax 315.453.9010 www.anchorgea.com

# **M**EMORANDUM

To: Bob Piniewski Date: March 3, 2011

Project Navigator

From: Chris Torell, Anchor QEA Project: 040284-01

**Cc:** David Keith and Elaine Darby

**Re:** Addendum to the Health and Safety Plan for Patrick Bayou Superfund Site

This memorandum is an addendum to the Health and Safety Plan for the Patrick Bayou Superfund Site, prepared specifically to support the Upstream Characterization Sampling and Analysis Plan sampling in several culverts at the Site. Although not anticipated, these culverts may constitute a confined space condition, consistent with 29 CFR 1910.146. As such, this addendum has been prepared as a conservative measure to support sediment sampling in these culverts. This addendum is supported by an Entry Permit for Permit-Required Confined Space (PRSC) (Attachment 1), which is required prior to initiating work in a culvert.

Provided below are Anchor QEA's confined space assessment and entry procedures. These, in conjunction with the permit, constitute Anchor QEA's HASP addendum to support the culvert sampling scope.

# **Confined Space Entry Procedures**

# **Purpose and Summary**

This procedure describes the requirements for identifying and working within confined spaces. It has been developed to ensure compliance with the OSHA Permit-Required Confined Spaces (PRCS) regulation (29 CFR 1910.146). Key provisions of this procedure include:

- PRCS
- Duties of PRCS Participants
- Permit System
- Training

- Non-Permit-Required Confined Spaces
- Rescue and Emergency Services
- Employee Access to Documentation
- Retention of Inspection and Test Logs
- Program Review

## **Definitions**

*Acceptable Entry Conditions* – The conditions that must exist in a permit space to allow entry so that employees involved with a PRCS entry can safely enter into and work within the space.

*Attendant* – An individual stationed outside one or more permit spaces who monitors the authorized entrants and who performs all attendant's duties described in this procedure.

*Authorized Entrant* – An employee who is authorized to enter a PRCS.

Blanking or Blinding – The absolute closure of a pipe, line, or duct, by the fastening of a solid plate (such as a spectacle blind or a skillet blind) that completely covers the bore and that is capable of withstanding the maximum pressure of the pipe, line, or duct, with no leakage beyond the plate.

#### *Confined Space* – A space that:

- Is large enough and so configured that an employee can bodily enter and perform assigned work
- Has limited or restricted means for entry or exit (for example, tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations)
- Is not designed for continuous employee occupancy

*Double Block and Bleed* – The closure of a line, duct, or pipe, by closing and locking or tagging two in-line valves and by opening and locking or tagging a drain or vent valve in the line between the two closed valves.

Engulfment – The surrounding and effective capture of a person by a liquid or finely divided (flowable) solid substance that can be aspirated to cause death by filling or plugging the respiratory system, or that can exert enough force on the body to cause death by strangulation, constriction, or crushing.

*Entry* – The action by which a person passes through an opening into a PRCS. Entry includes ensuing work activities in that space and is considered to have occurred as soon as any part of the entrant's body breaks the plane of an opening into the space.

*Entry Permit* – The written or printed document that is provided by the company to allow and control entry into a permit space.

*Entry Supervisor* – The person responsible for determining if acceptable entry conditions are present at a permit space where entry is planned, for authorizing entry, and overseeing entry operations, and for terminating entry as required by this section. An entry supervisor may also serve as an attendant or as an authorized entrant; as long as that person is trained and equipped for each role he/she fills.

*Hazardous Atmosphere* – An atmosphere that may expose employees to the risk of death, incapacitation, impairment or ability to self-rescue (that is, escape unaided from a permit space), injury, or acute illness, from one or more of the following causes:

- Flammable gas, vapor, or mist, in excess of 10 percent of its LEL.
- Airborne combustible dust at a concentration that meets or exceeds its LEL. NOTE: This concentration may be approximated as a condition in which the dust obscures vision at a distance of 5 feet or less.
- Atmospheric oxygen concentration below 19.5 percent or above 23.5 percent.
- Atmospheric concentration of any substance for which a dose or a published exposure guideline is available and which could result in employee exposure in excess of its dose or PEL.
- Any other atmospheric condition that is immediately dangerous to life or health (see Table 5-2).

*Hot Work Permit* – Written authorization to perform hot operations (for example, riveting, welding, cutting, burning, and heating) capable of providing a source of ignition.

*Immediately Dangerous to Life or Health (IDLH)* – Any condition that poses an immediate or delayed threat to life, would cause irreversible adverse health effects, or would interfere with an individual's ability to escape unaided from a confined space.

*Inerting* – The displacement of the atmosphere in a confined space by a noncombustible gas (such as nitrogen), to such an extent that the resulting atmosphere is noncombustible.

*Isolation* – The process by which a confined space is removed from service and completely protected against the release of energy and material into the space; by such means as blanking or blinding; misaligning or removing sections of lines, pipes, or ducts; a double block and bleed system; lockout or tagout of all sources of energy, including hydraulic or electric; blocking or disconnecting all mechanical linkages; or physically restraining moving parts.

*Line Breaking* – The intentional opening of a pipe, line, or duct, that is or has been carrying flammable, corrosive, or toxic material; an inert gas; or any fluid at a volume, pressure, or temperature capable of causing injury.

*Non-Permit-Required Confined Space* – A confined space that does not contain or, with respect to atmospheric hazards, have the potential to contain any hazard capable of causing death or serious physical harm.

*Oxygen-Deficient Atmosphere* – An atmosphere containing less than 19.5 percent oxygen by volume.

*Oxygen-Enriched Atmosphere* – An atmosphere containing more than 23.5 percent oxygen by volume.

*Permit-Required Confined Space (PRCS)* – A confined space that has one or more of the following characteristics:

- Contains or has a potential to contain a hazardous atmosphere
- Contains a material that has the potential for engulfing an entrant

- Has an internal configuration such that an entrant could be trapped or asphyxiated by inwardly converging walls or by a floor that slopes downward and tapers to a smaller cross section
- Contains any other recognized serious safety or health hazard

*Prohibited Condition* – Any condition in a PRCS that is not allowed by the permit during the period when entry is authorized.

*Rescue Service* – The personnel designated to rescue employees from PRCSs.

*Retrieval System* – The equipment (including a retrieval line, chest or full-body harness, wristlets, if appropriate, and a lifting device or anchor) used for non-entry rescue of persons from PRCSs.

*Testing* – The process by which the hazards that may confront entrants of a confined space are identified and evaluated. Testing includes specifying the tests that are to be performed in the confined space.

# Permit-Required Confined Spaces

Prior to beginning any PRCS entry operation, a PRCS entry permit will be completed by the entry supervisor. All such entries will be considered permit-required until or unless the space meets the definition of a non-permit-required confined space as previously defined. The following guidelines are to be followed for each PRCS entry:

- Combustible vapors will not exceed 10.0 percent of the LEL and oxygen levels must be between 19.5 and 23.5 percent by volume for entry to be allowed. Appropriate toxic gas/vapor action levels will also be established and documented on the permit.
- Lockout, tagout, tryout, and return to service procedures for potential sources of hazardous energy must be completed.
- As necessary, purging, inerting, flushing, or ventilating the space may be used to control hazardous atmospheres. Continuous mechanical ventilation will be used whenever entrants are in PRCSs that have or that can be expected to have a hazardous atmosphere.

- Inspecting, monitoring, and testing the PRCS to verify that acceptable conditions exist prior to and throughout the entry operation must be conducted. This includes: Conducting specific atmospheric tests as described on the entry permit. PRCSs will be tested as often as necessary to verify entrant safety, whenever operations or conditions change (e.g., temperature change or product agitation), and no less often than hourly.
- For confined spaces that cannot be completely isolated (e.g., sewers), continuous testing with real-time direct reading instruments is required.
- Testing for oxygen will occur first, followed by combustible gases, then toxic gases and vapors.
- PPE will be used as specified on the entry permit and will include:
  - Type of protective suits, boots, and gloves.
  - Type of face, head, and foot protection.
  - Type of harness (chest or full-body) with approved lifelines at least 1/2-inch in diameter, 2,000 pounds test, and meeting ANSI A10.14 requirements. The lifeline is to be attached at the center of the entrant's back near shoulder level, above the entrant's head, or at another point which presents a profile small enough for the successful removal of the entrant. (NOTE: Wristlets may be used only when a health and safety representative finds that a harness presents a greater hazard to the employee and wristlets are the safest, most effective alternative.) All lifelines will be secured to a mechanical extraction device or fixed point outside the PRCS. Mechanical extraction devices will be used for all vertical entries greater than 5 feet deep.
  - Type of respiratory protection, per the requirements of the site-specific HASP.
- MSDS will be readily available and provided to the medical facility when treating injured or exposed entrants.
- Lighting equipment required to safely illuminate the work will be utilized. NOTE:
   All lighting and electrical equipment used inside a PRCS will be of the appropriate
   National Electrical Code (NEC) rating. Rating should be Class I, Division I, unless the
   space specifically meets other rating class requirements.
- Protective barriers will be used to protect entrants from external pedestrian, vehicle, or equipment hazards.

- Ingress and egress equipment such as ladders or mechanical extraction devices will be used as necessary.
- Rescue and emergency services, procedures, and equipment will be determined prior to entry. The permit must specify whether the company or another source will provide these services and equipment, and how to summon them.
- Communication methods will be used that will provide continuous communication between entrants and attendants. This can be done using the standard system of lifeline "tugs" below, so long as the attendants continuously hold the lifelines in their hands:

#### Lifeline "Tug" Signals

1 Tug = Are you OK?

2 Tugs = Yes, I am OK.

3 Tugs = Exit the confined space immediately.

- An alternative communication system would be to provide all entrants and attendants with an air-powered horn. Substituting horn blasts for tugs, equivalent signals to the lifeline "tug" signals would be used. Any other or uncertain signals would require immediate exit.
- If these methods are not practical or possible, appropriately rated powered communication equipment will be provided.
- The number of attendants and other outside support personnel will be determined
  prior to entry. Each PRCS being entered will have a minimum of one dedicated
  attendant and one other support person (who may have other duties) within sight or
  call.

# **Duties of PRCS Participants**

Authorized entrants will:

- Know the hazards that may be faced during entry, including information on the mode, signs or symptoms, and consequences of potential exposures.
- Communicate with the attendant as necessary to enable the attendant to monitor entrant status and to enable the attendant to alert entrants of the need to evacuate the space.

- Alert the attendant whenever the entrant recognizes any warning sign or symptom of exposure to a dangerous situation, or if the entrant detects a prohibited condition.
- Exit from the permit space as quickly as possible whenever:
  - An order to evacuate is given by the attendant or the entry supervisor
  - The entrant recognizes any warning sign or symptom of exposure to a dangerous situation
  - The entrant detects a prohibited condition
  - An evacuation alarm is activated

### Attendants will:

- Know the hazards that may be faced during entry, including information on the mode, signs or symptoms, and consequences of potential exposures.
- Be cognizant of possible behavioral effects of hazard exposure in authorized entrants.
- Continuously maintain an accurate count of authorized entrants in the PRCS so that
  the means used to identify authorized entrants accurately identifies who is in the
  permit space.
- Remain outside the PRCS during entry operations until relieved by another attendant.
- Communicate with authorized entrants as necessary to monitor entrant status and to alert entrants of the need to evacuate the space.
- Monitor activities inside and outside the space to determine if it is safe for entrants to remain in the space, and order the authorized entrants to evacuate the PRCS immediately under any of the following conditions:
  - If the attendant detects a prohibited condition
  - If the attendant detects the behavioral effects of hazardous exposure in an authorized entrant
  - If the attendant detects a situation outside the space that could endanger the authorized entrants
  - If the attendant cannot effectively and safely perform all prescribed duties
- Summon rescue and other emergency services as soon as the attendant determines that authorized entrants may need assistance to escape from the PRCS.
- Take the following actions when unauthorized persons approach or enter a PRCS while entry is underway:

- Warn the unauthorized persons that they must stay away from the PRCS.
- Advise the unauthorized persons that they must exit immediately if they have entered the PRCS.
- Inform the authorized entrants and the entry supervisor if unauthorized persons have entered the PRCS
- Perform non-entry rescues.
- Perform no duties that might interfere with the attendant's primary duty to monitor and protect the authorized entrants.

### Entry supervisors will:

- Remain immediately available on-site throughout entry operations.
- Know the hazards that may be faced during entry, including information on the mode, signs and symptoms, and consequences of potential exposures.
- Verify, by checking that the appropriate entries have been made on the permit, that all tests specified by the permit have been conducted, and that all procedures and equipment specified by the permit are in place before endorsing the permit and allowing entry to begin.
- Terminate the entry and cancel the permit as required.
- Verify that qualified rescue services are available and that the means for summoning them are operable. Evaluate capabilities of service prior to entry.
- Arrange for the removal of unauthorized individuals who enter or who attempt to enter the PRCS during entry operations.
- Determine when responsibility for a permit space entry operation is transferred and at intervals dictated by the hazards and operations performed within the space, and that entry operations remain consistent with the terms of the entry permit, and acceptable entry conditions are maintained.
- Document on the entry permit any incidents or circumstances requiring review of the confined space entry program. Such incidents may include:
  - Unauthorized entry
  - The detection of a condition or hazard not authorized by the permit
  - The occurrence of an injury or near-miss during entry
  - A change in use or configuration of the space
  - Employee complaints about the program

 Dictate procedures for coordination of entry when personnel from multiple employers will work simultaneously within a PRCS.

### Permit System

Before entry can be authorized, the entry supervisor must complete and sign an entry permit to document that all pre-entry requirements have been met and that acceptable entry conditions exist. The completed permit will be posted at the primary entrance to the PRCS, and made available to each employee entering the space, or to that employee's authorized representative.

All entry permits are valid for a maximum of one work shift, and will be canceled by the entry supervisor when the shift ends, PRCS operations are complete, or whenever a prohibited condition arises in or near the space. All PRCSs will be securely closed or barricaded whenever the entry permit is canceled.

Supplemental information regarding the location of each entrant will be provided as described below:

- The current entry status of all entrants will be logged, with new entries made whenever the entry status of an entrant changes.
- Each entrant will securely affix a tag bearing their name to the outside lifeline fitting, which is attached to a secure point.

### **Training**

Prior to assignment to PRCS entry work, all affected employees will receive training in the hazards of confined spaces, work practices to control these hazards, and duties to be performed. Training will consist of a detailed review of this procedure, as well as the hazards inherent with the particular PRCS that will be entered.

Confined space entry training for entrants or attendants can be conducted by a qualified entry supervisor or a health and safety representative. Confined space entry training for entry supervisors must be conducted by a health and safety professional.

The company will maintain confined space entry training records to include employee name and signature, date of training, and signature of the trainer.

### Non-Permit-Required Confined Spaces

All confined spaces initially considered PRCSs can be reclassified as non-permit-required confined spaces by the entry supervisor only under the following conditions:

- All contaminants have been isolated or removed.
- All actual or potential atmospheric hazards have been eliminated, with testing verification.
- Ventilation is not required to maintain control of atmospheric hazards.
- All recognized hazards, including engulfment, within the space have been eliminated.
- The space will be re-evaluated (and reclassified to permit-required, if needed) whenever the use or configuration of the space changes in any way that might increase the hazards to the entrants. All entrants will exit the space immediately when hazards are noted.
- The entry supervisor will make the certification that all hazards have been removed on the entry permit.
- The entry permit will be posted at the entrance to the confined space.

## Rescue and Emergency Services

The company recommends the use of non-company rescue services whenever possible. In certain instances, such as unavailability of a qualified outside provider, company employees can participate in rescues if they have been provided the required equipment and training.

#### Outside Rescue Services

Prior to designating a non-company rescue service, an evaluation of their capabilities must be conducted. This documented evaluation can be conducted by an entry supervisor or a health and safety representative. The rescue service must be certified by the evaluator as capable of performing rescues prior to being identified as the rescue service provider.

Each selected rescue service will be informed of the hazards they may encounter at the location. They will also be provided access to all PRCSs from which a rescue may be necessary.

### Company Rescue Services

Company personnel assigned to provide emergency entry and rescue services will be trained annually in the proper use of PPE and rescue equipment. Such training will include a simulated rescue exercise.

Company rescue services will be evaluated and certified by the evaluator as capable of performing rescues prior to being identified as the rescue service provider.

### **Employee Access to Documentation**

Each employee participating in a PRCS entry, or that employee's authorized representative, will be provided an opportunity to observe all testing and be provided a copy of the testing results. Each employee, or that employee's authorized representative, may also request the company to re-evaluate the PRCS because the employee or representative has reason to believe that the evaluation of the space may not have been adequate.

### Retention of Inspection and Test Logs

A copy of all entry permits and other documents related directly to the PRCS entry (e.g., hot work permits, air monitoring records, etc.) will be maintained in project files. If requested, these documents will also be made available to all employees participating in a PRCS entry, or to their authorized representatives.

### Program Review

The health and safety representative responsible for each location performing PRCS operations will review all entry permits for incidents or problems occurring during entry annually. Incidents or problems may include injuries, accidents, unauthorized entries, or any other event that indicates that improvements can be made in the PRCS program.

# ENTRY PERMIT FOR PERMIT-REQUIRED CONFINED SPACE (PRCS)

Project/Location	Project No			
Location of PRCS	Identity of PRCS			
Describe Hazards of PRCS (Chemical and I	Physica	al)		
,	•	•		
Purpose This Permit Authorized				
CHECKLIST	YES	DOES NOT APPLY	PERSONAL PROTECTIVE EQUIPMENT (Circle)  EYE/FACE Chemical Goggles Face Shield Safety Glasses	
All lines leading to and from the space have been blinded or disconnected.			EXTREMITIES Hard Hat Hoods Boot Covers	
Electrical service disconnected or locked out.			Gloves (Material)	
All grounding and bonding cables in place.			Boots (Material)	
All lighting, fittings, power equipment, and extension cords are rated for anticipated atmosphere.				
Ground Fault Circuit Interrupter (GFCI) checked and functioning.			RESPIRATORY   SCBA   Supplied Air   Egress System	
All ignition sources have been isolated.			Air Purifying (Cartridge)	
All respiratory equipment and alarms checked and functional			Powered Air Purifying (Cartridge)	
All safety harnesses and lifelines checked.			OTHER Hearing Protection Harness & Lifeline	
All required PPE checked and in use.			Chest or Parachute	
Have all entrants, attendants, and entry supervisors received appropriate training?			RESCUE EQUIPMENT Mechanical Extraction Device	
Attendant(s) trained in non-entry rescue procedures.			First Aid Kit SCBA Other (Specify)	
Rescue service has been identified and will be available for entry rescue.				
Has rescue service passed evaluation?			COMMUNICATION METHOD Lifeline "Tug" Signals	
Appropriate rescue equipment available and checked.			Air-powered Horn Signals	
Mechanical ventilation system in use and effective.			Other	
All tests have been completed and indicate that entrance requirements have been met.				
Appropriate warning signs have been posted and unauthorized personnel have been excluded from the PRCS.				
IF ANSWER TO ANY OF THE ABOVE QUESTIONS I NOT PERMITTED.	S <i>NO</i> , E	NTRY IS		
OTHER PERMITS ISSUED FOR WORK IN PRCS:				
OTHER HAZARD CONTROL PROCEDURES OR INSTR	UCTION	NS:		
RESCUE PROCEDURES:				

# TEST DATA OXYGEN, FLAMMABILITY, AND TOXIC CONTAMINANT(S)

Time	Percent	Percent LEL	(0.1	(0.11 )	(0.1	(0.11 )	(0.11 )	Tester's Initials	Comments	
	Oxygen	LEL	(Other)	(Other)	(Other)	(Other)	(Other)	initials		
	'S SIGNAT		•				<u> </u>			
AU	J <b>THORIZE</b>	D ENTRA	NTS					AUTHOL	RIZED ATTENDANT(S)	
								RES	CUE PERSONNEL	
							.			
Diagram	the confin	ad anasa in	dianta lana	tion of mor	nreare and	zantilatan	a Indicate	location(a)	whom tosts conducted	
Diagran	i the confin	ed space in	aicate ioca	uon oi ma	nways and	venmators	s. maicate	iocation(s)	where tests conducted.	
									) ( Man-way	
									∞ Ventilator	
									X Test Location	
	TABLE EN			2 0	1 ,	10.5 1.2	22.50/ 2	G 1 1	11 11 100/ 157	
	ry Permit co missible Lev				gen betweer	1 19.5 and 2			ble gases below 10% LEL	
1. 101	missione Le	veis of toxic	e gases (nst	,.			5. 0			
			<del></del> -							
DD CC C	AFE FOR I	ENI/DDX/								
Date/Tin		ZNIKY /								
	Entry Super					Cianatu	180			
						Signatu	ire			
	Entry Superv									
Entry Pe	rmit Expires	(no longer	than 1 shift	t): I	Date/Time		/			
ENTRY	PERMIT (	CANCELE	D							
Date/Tin	ne	/				Signature				
Reason (	Reason ( $$ ) Work Complete Authorized Conditions Not Met Incident									
PROBL.	EMS DURI	NG ENTR	Y AND RI	ESOLUTIO	N. Please	Describe:				
TROBE	LIVIO DOINI	I (G LI (II)		25020110	Ji i i icuse	Describe.				
RECLA	SSIFICATI	ON TO N	ON-PERM	IT-REOU	IRED CON	IFINED SI	PACE			
	hazard rem									
TESTIN	G VERIFIC	ΔΤΙΩΝ ΩΊ	OWN AT	гімғ		ON	TEST DAT	'А СИЛОТ	AROVE	
DATE/T		/ /	OWN AI.		SUPERVIS			ACHARI	ADU VE.	
	VED BY:	·								
		Healtl	h and Safety	Represent	ative Signa	ture		D	ate	

# ATTACHMENT 2 FIELD FORMS



# **Sediment Probing Form**

Station	Date	Time	Coordir		Water Depth	Depth of Refusal	Sediment Thickness	Sediment Type <sup>b</sup>	Comments
Station	Date		Latitude	Longitude	(cm)	(cm)	(cm)	Sediment Type	Commence

Recorded by	•

a - Coordinates to be recorded in North American Datum 1983 (NAD 83)

b - Sediment Type - muddy (cohesive) bed, sandy (non-cohesive) bed, rocky bed - based on resistance met by pole



### **Surface Sediment Field Sample Record**

		<u>Pro</u>	ject No:		Station I	D:	
Sampling Crew:	:						
	:			Sampling Method:			
Sampling Vessel:				, ,			
Subcontractor(s):			_	Weather:			
Station Coordinates:	•			_			
Cialion Coordinates.				_			
_	E / Long.			_			
Datum:	NAD 83 / WGS 84		zone:				
Sample ID:				_			
Analysis:	Metals / TBT / SVOC			Other:			
	TS / TVS / Grain Size			Other:			
	(Circle Appropriate A	lalyses					
Grab Number:	Water Depth:	ft.		Grab Recovery:_	c	m Time: _	
	Tide Level:			Sample Interval:_	c	em	
Bioassay / Chemistry	Depth MLLW:	ft.	1	<u></u>		T.	<u> </u>
Sediment Type:	Sediment Color:		Density:	Sediment Odor:		Sheen:	Moisture:
cobble	D.O.		Very soft/Loose	none	H2S	none	Dry
gravel	gray		soft/loose	slight	Petroleum	trace	Damp
sand C M F	black		mod dense/stiff	moderate	other:	slight	Moist
silt clay organic matter	brown brown surface		dense/stiff very dense/stiff	strong overwhelming		moderate	Wet
Comments:	biowii suiiace		very dense/sun	overwheiming		heavy	
Grab Number:	Water Depth:			Grab Recovery:_ Sample Interval:_			
Bioassay / Chemistry	Depth MLLW:	ft.					
Sediment Type:	Sediment Color:		Density:	Sediment Odor:		Sheen:	Moisture:
cobble	D.O.		Very soft/Loose	none	H2S	none	Dry
gravel	gray		soft/loose	slight	Petroleum	trace	
sand C M F	black		I I /. Cff				Damp
	- Idok		mod dense/stiff	moderate	other:	slight	Moist
silt clay	brown		dense/stiff	strong	other:	moderate	
silt clay organic matter					other:	•	Moist
silt clay organic matter	brown		dense/stiff	strong	other:	moderate	Moist
silt clay organic matter	brown		dense/stiff	strong	other:	moderate	Moist
silt clay	brown		dense/stiff	strong	other:	moderate	Moist
silt clay organic matter Comments:	brown	ft.	dense/stiff	strong overwhelming Grab Recovery:_		moderate heavy	Moist
silt clay organic matter  Comments:  Grab Number:	brown brown surface  Water Depth: Tide Level:	ft.	dense/stiff	strong overwhelming		moderate heavy	Moist Wet
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry	brown brown surface  Water Depth: Tide Level: Depth MLLW:	ft.	dense/stiff very dense/stiff	strong overwhelming  Grab Recovery:_ Sample Interval:_		moderate heavy m Time: _	Moist Wet
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type:	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color:	ft.	dense/stiff very dense/stiff  Density:	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor:		moderate heavy  m Time: m Sheen:	Moist Wet
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type: cobble	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color: D.O.	ft.	dense/stiff very dense/stiff  Density: Very soft/Loose	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor: none	c c	moderate heavy  m Time: cm  Sheen: none	Moist Wet  Moisture: Dry
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type: cobble	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color:	ft.	dense/stiff very dense/stiff  Density:	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor:		moderate heavy  m Time: m Sheen:	Moist Wet
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type: cobble gravel	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color: D.O.	ft.	dense/stiff very dense/stiff  Density: Very soft/Loose	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor: none	c c	moderate heavy  m Time: cm  Sheen: none	Moist Wet  Moisture: Dry
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type: cobble gravel sand C M F silt clay	Water Depth: Tide Level: Depth MLLW: Sediment Color: D.O. gray	ft.	dense/stiff very dense/stiff  Density: Very soft/Loose soft/loose	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor: none slight moderate strong	H2S Petroleum	m Time: cm Sheen: none trace	Moist Wet  Moisture: Dry Damp
silt clay organic matter	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color: D.O. gray black	ft.	Density: Very soft/Loose soft/loose mod dense/stiff	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor: none slight moderate	H2S Petroleum	moderate heavy  m Time: cm  Sheen: none trace slight	Moist Wet  Moisture: Dry Damp Moist
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type: cobble gravel sand C M F silt clay organic matter	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color: D.O. gray black brown	ft.	Density: Very soft/Loose soft/loose mod dense/stiff dense/stiff	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor: none slight moderate strong	H2S Petroleum	moderate heavy  m Time: cm  Sheen: none trace slight moderate	Moist Wet  Moisture: Dry Damp Moist
silt clay organic matter  Comments:  Grab Number:  Bioassay / Chemistry  Sediment Type: cobble gravel sand C M F silt clay	brown brown surface  Water Depth: Tide Level: Depth MLLW: Sediment Color: D.O. gray black brown	ft.	Density: Very soft/Loose soft/loose mod dense/stiff dense/stiff	strong overwhelming  Grab Recovery: Sample Interval: Sediment Odor: none slight moderate strong	H2S Petroleum	moderate heavy  m Time: cm  Sheen: none trace slight moderate	Moist Wet  Moisture: Dry Damp Moist

Date/Time Lab Drop Off:

Recorded	by	

# Visual Classification of Subsurface Core



Job No.     Core Pushed By       Exploration No.     Core Logged By       Core No.     Type of Core	I = Ia	QEA SEE
Core No.  Water Depth/Elevation of Core Cored Length (feet; from log)  Core Recovery (feet)  Core Recovery (feet)  Core Recovery (feet)  Core Core Logged By  Type of Core Shelby Piston Core Other  Diameter of Core (inches)  Core Quality Good Fair Poor Disturbed  Average % Compaction =  Classification and Remarks (Color, Consistency, Moisture, Grain Size, Sheen, Odor)		Date
Type of Core   Shelby   Piston Core   Other		
Type of Core   Shelby   Piston Core   Other	Exploration No.	Core Logged By
Water Depth/Elevation of Core  Cored Length (feet; from log)  Core Recovery (feet)  Depth  Depth  Units  ()  Sample  Analytes  Diameter of Core (inches)  Core Quality  Good  Fair  Poor  Disturbed  Average % Compaction =  Classification and Remarks  (Color, Consistency, Moisture, Grain Size, Sheen, Odor)	Core No.	
Core Recovery (feet)  Core Recovery (feet)  Core Quality Good Fair Poor Disturbed  Average % Compaction =  Classification and Remarks (Color, Consistency, Moisture, Grain Size, Sheen, Odor)	Water Depth/Elevation of Core	Diameter of Core (inches)
Core Recovery (feet)  Average % Compaction =    Option	Cored Length (feet: from log)	Core Quality
Depth units (Color, Consistency, Moisture, Grain Size, Sheen, Odor)		Average 0/ Compaction
	Core Recovery (reet)	Average % Compaction =
	` '	

# Water Quality Sample Form



Project Name:	Project Number:	
6 11 0		
Sampling Crew:	Weather:	
Subcontractor(s):	<del></del>	
Date:	Consulting Marklands	
Station Coordinates:	Sampling Method:	
	<del></del>	
Station ID:		
Sample ID:		
Time:		
Water Depth:		
Sample Depth:		
Station ID:		
Station ID:		
Sample ID:		
Motor Dooth		
Water Depth: Sample Depth:		
запре верш.		
Station ID:		
Camanda ID.		
Water Depth:		
Sample Depth:		
Station ID:		
Sample ID:		
Time:		
Water Depth:		
Sample Depth:		
Comments		
Comments:		
		_

# **PCB Field Assay Bench Sheet**

1 %	ANCHOR
K	ANCHOR QEA :::

Date:	
Project:	
Project No:	
Analyst(s):	

Reagent/ Standard:	Lot#	Expiration

Run	_	Sample	Dilution	Absorbance	Concentration	
No.	Sample ID	Weight (g) <sup>a</sup>	Factor	(nm)	(mg/kg) <sup>b</sup> Comme	Comments
1	Negative Control Rep.1				. 0. 0.	
2	Negative Control Rep.2					
3	Standard 1 Rep.1					
4	Standard 1 Rep.2					
5	Standard 2 Rep.1					
6	Standard 2 Rep.2					
7	Standard 3 Rep.3					
8	Standard 3 Rep.4					
9	Control Sample					
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
а	Recommended dry weight to achieve	detection limits = 1	0g ± 0.1g			
b	Detection limit = 0.5 ppm; upper limit	t = 10 ppm; sample v	weight and dilut	ion factor will affect	achievable range	

Additional Notes:

# ATTACHMENT 3 RAPID ASSAY TEST KIT USERS GUIDE

### STRATEGIC DIAGNOSTICS INC.

# RaPID Assay® PCB Test Kit A00133/A00134

### **Intended Use**

The RaPID Assay® PCB Test Kit can be used as a quantitative, semi-quantitative or qualitative enzyme immunoassav (EIA) for analysis the (polychlorinated biphenyl) in water (groundwater, surface water, well water). For applications in other matrices please contact our Technical Service department or refer to the soil application procedure provided. The RaPID Assay® PCB Test Kit allows reliable and rapid screening for PCB (measured and reported as Aroclor 1254), with quantitation between 0.5 and 10 ppb (as Aroclor 1254), in water. The minimum detection level of the kit is 0.2 ppb (as Aroclor 1254.)

### **Test Principles**

The PCB RaPID Assay® kit applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of PCB and related compounds. The sample to be tested is added, along with an enzyme conjugate, to a disposable test tube, followed by paramagnetic particles with antibodies specific to PCB attached. Both PCB (which may be in the sample) and the enzyme labeled PCB (the enzyme conjugate) compete for antibody binding sites on the magnetic particles. At the end of an incubation period, a magnetic field is applied to hold the paramagnetic particles (with PCB and labeled PCB analog bound to the antibodies on the particles, in proportion to their original concentration) in the tube and allow the unbound reagents to be decanted. After decanting, the particles are washed with Washing Solution.

The presence of PCB is detected by adding the enzyme substrate (hydrogen peroxide) and the chromogen (3,3',5,5' – tetramethylbenzidine). The enzyme labeled PCB analog bound to the PCB antibody catalyzes the conversion of the substrate/chromogen mixture to a colored product. After an incubation period, the reaction is stopped and stabilized by the addition of acid. Since the labeled PCB (conjugate) was in competition with the unlabeled PCB (sample) for the antibody sites, the color developed is inversely proportional to the concentration of PCB in the sample.

**NOTE**: Color development is inversely proportional to the PCB concentration.

Darker color = lower concentration Lighter color = higher concentration

The determination of the PCB level in an unknown sample is interpreted relative to the standard curve generated from kit standards after reading with a spectrophotometer.

### Performance Characteristics

The PCB RaPID Assay® will detect different PCB Aroclors to different degrees. Refer to the table below for data on several of these. The PCB RaPID Assay® kit provides screening results. As with any analytical technique (GC, HPLC, etc. ) positive results requiring some action should be confirmed by an alternative method.

The PCB RaPID Assay® immunoassay test does not differentiate between PCB and other related compounds. The table below shows compounds at the method detection limit (MDL) which is the lowest concentration of the compound, in water, that can be picked up in the assay. The limit of quantitation (LOQ) is an approximate concentration, in water, required to yield a positive result at the lowest standard. This is the lowest concentration of the compound that can be quantified in the assay. The IC50 is the concentration required to, inhibit one half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

)

Compound	MDL	LOQ	IC50
_	(ppb)	(ppb)	(ppb)
Aroclor 1254	0.20	0.50	3.6
Aroclor 1260	0.20	0.32	2.3
Aroclor 1248	0.22	0.59	4.22
Aroclor 1242	0.34	1.22	8.8
Aroclor 1262	0.36	0.66	4.74
Aroclor 1232	0.84	2.61	18.76

Aroclor 1268	0.92	3.03	21.80
Aroclor 1016	0.94	3.56	25.60
Aroclor 1221	13.54	22.58	162.60

\*The following compounds demonstrated no reactivity in the PCB RaPID Assay® test kit at concentrations up to 10,000 ppb: Biphenyl, 2,5-Dichlorophenol, 2,3,5-Trichlorophenol, Di-n-octyl-phthalate.

The presence of the following substances up to 250 ppm were found to have no significant effect on PCB RaPID Assay® results: copper, nickel, zinc, mercury, manganese, phosphate, sulfate, sulfite, magnesium, calcium, nitrate and thiosulfate. Humic acid up to 25 ppm and iron to 100 ppm were found to have no significant effect. In addition, sodium chloride concentrations up to 1.0 M showed no effect on results.

### **Precautions**

- Training is strongly recommended prior to using the RaPID Assay® test system. Contact Strategic Diagnostics for additional information.
- Treat PCB, solutions that contain PCB, and potentially contaminated samples as hazardous materials.
- Use gloves, proper protective clothing, and methods to contain and handle hazardous material where appropriate.
- Reagents must be added in a consistent manner to the entire rack. A consistent technique is the key to optimal performance. Be sure to treat each tube in an identical manner.
- Water samples should be at a neutral pH prior to analysis. Samples containing gross particulate should be filtered (e.g. 0.2 um Anotop<sup>TM</sup> 25 Plus, Whatman, Inc.) to remove particles.
- Store all test kit components at 2°C to 8°C (36°F to 46°F). Storage at ambient temperature (18°C to 27°C or 64°F to 81°F) on the day of use is acceptable. Test tubes require no special storage and may be stored separately to conserve refrigerator space.
- Allow all reagents to reach ambient temperature (18°C to 27°C or 64°F to 81°F) before beginning the test.
  This typically requires at <u>least</u> 1 hour to warm from recommended storage conditions.

- Do not freeze test kit components or expose them to temperatures above 100°F (39°C).
- Do not use test kit components after the expiration date.
- Do not use reagents or test tubes from one test kit with reagents or test tubes from a different test kit.
- Do not mix reagents from kits of different lot numbers.
- Use approved methodologies to confirm any positive results.
- Do not under any circumstances attempt to disassemble the base of the magnetic rack. Magnets will be violently attracted to each other.
- Adequate sample number and distribution are the responsibility of the analyst.
- The photometer provided in the accessory kit requires electricity and comes with a 110V adapter. Adapters for 220V are available. Do not attempt to operate with a car adaptor.
- Do not expose color solution to direct sunlight.
- Do not dilute or adulterate test reagents or use samples not called for in the test procedure; this may give inaccurate results.
- Tightly recap the standard vials when not in use to prevent evaporative loss.

### **Materials Provided**

• Antibody Coupled Paramagnetic Particles in buffered saline containing preservative and stabilizers.

30 test kit: one 20 mL vial 100 test kit: one 65 mL vial

Enzyme Conjugate.

30 test kit: one 10 mL vial 100 test kit: one 35 mL vial

Standards

Three concentrations (0.25, 1.0 and 5.0 ppb) of PCB standards (as Aroclor 1254) in buffered saline

containing preservative and stabilizers are supplied. Each vial contains 4 mL.

Control

A concentration (approximately 3 ppb) of PCB (as Aroclor 1254) in buffered saline containing preservative and stabilizers. A 4 mL volume is supplied in one vial.

• Diluent/Zero Standard

Buffered saline containing preservative and stabilizers without any detectable PCB.

30 test kit: one 10 mL vial 100 test kit: one 35 mL vial

• Color Solution containing hydrogen peroxide and 3,3',5,5'-tetramethylbenzidine in an organic base.

30 test kit: one 20 mL vial 100 test kit: one 65 mL vial

• Stop Solution containing a solution of 2M sulfuric acid.

30 test kit: one 20 mL vial 100 test kit: one 60 mL vial

Washing Solution containing preserved deionized water.

30 test kit: one 70 mL vial 100 test kit: one 250 mL vial

• Polystyrene test tubes

30 test kit: one 36 tube box 100 test kit: three 36 tube boxes

• User's Guide

# Materials Required and Ordered Separately

See "Ordering Information" for the appropriate catalogue numbers.

### Rapid Assay® Accessory Kit

Accessory equipment may be rented or purchased from Strategic Diagnostics. See "Ordering Information" for the appropriate catalogue numbers.

The accessory kit contains the following items:

Adjustable Volume Pipet

- Eppendorf<sup>TM</sup> Repeater<sup>®</sup> Pipettor
- Electronic timer
- Portable balance capable of weighing 10 g (for soil samples)
- Vortex mixer
- Magnetic separation rack
- RPA-I RaPID Analyzer (or equivalent spectrophotometer capable of reading 450 nm in a 1 mL sample size).

### Other Items

- 12.5 mL Combitips® for the Repeater pipettor for 0.25 mL to 1.25 mL dispensing volumes (5)
- Pipet tips for adjustable volume pipet (100-1000 uL)

**NOTE:** Order replacement Combitips<sup>®</sup> and pipet tips separately. See the "Ordering Information" section.

### Materials Required but Not Provided

- Methanol (HPLC grade or equivalent) for water analysis
- Protective clothing (e.g., latex gloves)
- Absorbent paper for blotting test tubes
- Liquid and solid waste containers
- Marking pen
- Instructional video (optional)

## **Suggestions for Pipettor Use**

- Practice using both pipettes (adjustable volume and Repeater pipettor) with water and extra tips before you analyze your samples.
- Use a new tip each time you use the Repeater pipettor to pipette a different reagent to avoid reagent cross-contamination. Tips can be rinsed thoroughly, dried completely and reused. By using the same tip to dispense the same reagent each time you can avoid cross contamination.

NOTE: Repeator tips should be changed periodically (after ~10 uses) since precision deteriorates with use.

- Draw the desired reagent volume into the Repeater pipettor and dispense one portion of the reagent back into the container to properly engage the ratchet mechanism. If you do not do this, the first volume delivered may be inaccurate.
- To add reagents using the Repeater pipettor, pipette down the side of the test tube just below the rim.
- When adding samples and standard using the positive displacement pipettor, always pipette into the bottom of the tube without touching the sides or bottom of the tube.
- Use a new adjustable volume pipet tip each time you pipette a new unknown.

### **Assay Procedure**

Prior to performing your first Rapid Assay®, please take time to read the package inserts in their entirety and review the videotape if available. On site training is strongly recommended for new users of this test system. Please contact your account manager for further information. This procedure is designed for quantitative analysis. For running the kit semi-quantitatively or qualitatively, please contact Technical Support.

### Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

1. Water samples should be collected in glass vessels with teflon cap liners). Immediately upon collection, water samples should be diluted with an equal volume (1:1) of methanol (HPLC grade) to prevent adsorptive losses to the glass containers. This is a 2x dilution, which must be accounted for when interpreting results. See "Results Interpretation", Section 3a for further details. Use this diluted sample as "sample" in "Perform the Test".

# NOTE: This 2x dilution is <u>not</u> required for soil samples.

- 2. Samples should be collected in appropriately sized and labeled containers.
- 3. If testing soil samples, follow the SDI Sample Extraction Kit User's Guide or the appropriate technical bulletin to properly collect and store your sample.

4. Samples should be tested as soon as possible after collection. If this is not possible, storage at 4°C (39°F) is recommended to minimize evaporative losses.

### Set Up

- 1. Remove kits from refrigerator. All reagents must be allowed to come to room temperature prior to analysis. Remove reagents from packaging and place at room temperature <u>at least</u> 1 hour prior to testing.
- 2. Turn on the RPA-1 or other spectrophotometer. The RPA-1 should be warmed up for at least 30 minutes prior to the run.
- 3. Label five 12.5 mL Combitips "Conjugate", "Particles", "Wash", "Color" and "Stop". In addition, add the name of the compound you are testing for to each Combitip.
- Remove nine clean blank test tubes for standards and control and one test tube for each sample (if testing in singlicate). Label the test tubes according to contents as follows.

•	
Tube #	<u>Contents</u>
1	Negative control (replicate 1)
2	Negative control (replicate 2)
3	Standard 1 (replicate 1)
4	Standard 1 (replicate 2)
5	Standard 2 (replicate 1)
6	Standard 2 (replicate 2)
7	Standard 3 (replicate 1)
8	Standard 3 (replicate 2)
9	Control
10	Sample 1
11	Etc.

\*Label at top of tubes to avoid interference with reading of tubes in photometer

### Sample Extraction, Filtration and Dilution

Filtration may be necessary to remove gross particulate from the water sample. If testing samples at levels higher than standard kit level is desired, contact SDI for special instructions. Water samples should be diluted 1:1 in methanol as described in "Collect/Store the Sample". Please follow the instructions from the SDI Sample Extraction Kit to prepare and dilute the soil extract prior to running the assay.

RaPID Assay PCB Test Kit 5

### Perform the Test

1. Separate the upper rack from the magnetic base. Place labeled test tubes into the rack.

 Add 200 uL of standards, control or samples to the appropriate tubes using the adjustable volume pipet with the dial set on 0200. The negative control, standards and control must be run with each batch of samples.

NOTE: Sample should be added to the bottom of the tube by inserting the pipet tip into the tube without touching the sides or the bottom of the tube. Take care not to contact sample with pipette tip once dispensed into bottom of the tube.

- 3. Using the Repeater Pipettor with the "Conjugate" tip attached and the dial set on "1", add 250 uL of Enzyme conjugate down the **inside wall** of each tube. (Aim the pipet tip ½" to ½" below the tube rim or tube wall; deliver liquid gently to avoid splashback.)
- 4. Thoroughly mix the magnetic particles by swirling (avoid vigorous shaking) and attach the "Particles" tip to the Repeater Pipettor. With the dial set on "2" add 500 uL of magnetic particles to each tube, aiming down the side of the tube as described above. Vortex, mixing each tube 1 to 2 seconds at low speed to minimize foaming. Pipetting of magnetic particles should be kept to 2 minutes or less.
- 5. Incubate 15 minutes at room temperature.
- 6. After the incubation, combine the upper rack with the magnetic base and press all tubes into the base; allow 2 minutes for the particles to separate.
- 7. With the upper rack and magnetic base combined, use a smooth motion to invert the combined rack assembly over a sink and pour out the tube contents.

NOTE: If the rack assembly inadvertently comes apart when lifting to pour out tube contents, recombine and wait an additional 2 minutes to allow particles to separate.

8. **Keep the rack inverted** and gently blot the test tube rims on several layers of paper towels. It is important to remove as much liquid as possible but **do not bang** the rack or you may dislodge the magnetic particles and affect the results.

9. Set the Repeater Pipettor dial to "4" and put on the tip labeled "Wash". Add 1 mL of Washing Solution down the inside wall of each tube by using the technique described earlier. Vortex tubes for 1-2 seconds. Wait 2 minutes and pour out the tube contents as described previously. Repeat this step one more time.

# NOTE: The number of washes and wash volume are important in ensuring accurate results.

- 10. Remove the upper rack (with its tubes) from the magnetic base. With the "Color" tip attached to the Repeater Pipet and the dial set to "2" add 500 uL of Color Reagent down the inside wall of each tube as described previously. Vortex 1 to 2 seconds (at low speed).
- 11. Incubate 20 minutes at room temperature. During this period, add approximately 1 mL of Washing solution to a clean tube for use as an instrument blank for "Results Interpretation".
- 12. After the incubation, position the Repeater pipettor at Setting "2" and use the "Stop" tip to add 500 uL of Stop solution to all test tubes.
- 13. Proceed with results interpretation.

WARNING: Stop solution contains 2M sulfuric acid. Handle carefully.

### Results Interpretation

- 1. After addition of Stop Solution to the test tubes, results should be read within 15 minutes.
- 2. Wipe the outside of all antibody coated tubes prior to photometric analysis to remove fingerprints and smudges.

#### Photometric Interpretation Using the RPA-I

1. The RPA-I photometer (provided in the Rapid Assay® Accessory kit) can be used to calculate and store calibration curves. It is preprogrammed with various RaPID Assay® protocols. For the PCB RaPID Assay® test kit, parameter settings are as follows:

Data Reduct:

Lin. Regression

Xformation:

Ln/LogitB

Read Mode:

Absorbance

Wavelength:

450 nm

Units

PPB

# Rgt Blk :

0

Calibrators:

# of Cals

4

# of Reps :

2

### Concentrations:

#1

0.00 ppb

#2

0.25 ppb

#3

1.00 ppb

#4

5.00 ppb

Range

- Tr

Correlation :

0.10 - 5.00 0.990

Rep. %CV:

10%

NOTE: Prior to analysis the RPA-I User's Manual should be thoroughly reviewed for more detailed operation instructions.

2. Follow the instrument prompts to read the absorbance of all tubes:

Instrument Display	Operator Response
SELECT COMMAND RUN PROTOCOL	Press RUN Scroll using the YES [] or NO [] keys until the desired protocol appears. Then press ENTER
SPL. REPLICATES (1-5)	Press 1 (for analysis of samples in singlicate.) Press ENTER
BLANK TUBE,	Insert blank tube
INSERT TUBE,	containing 1mL wash
EVALUATING TUBE,	solution.
REMOVE TUBE (Beep)	Remove tube
CAL #1, REP. #1, INSERT TUBE,	Insert Tube #1

EVALUATING TUBE,

REMOVE TUBE (Beep)

Remove tube

Follow prompts to read tubes.

**NOTE:** Tube order is important. The RPA-I expects to see the standards in ascending order, in duplicate, starting with the negative control.

Following evaluation of all standards, the instrument will display:

PRINTING DATA,	Data will print
PRINTING CURVE	Curve will print only if programmed to print (See RPA1 User's Manual).
CTRL #1 REP #1, INSERT TUBE, EVALUATING TUBE,	Insert Control Tube
REMOVE TUBE (Beep)	Remove Tube
EDIT CALIBRATORS YES/NO	Press NO (if editing is necessary press YES and refer to the RPA1 User's Manual).
SPL #1 REP#1 INSERT TUBE EVALUATING TUBE <sup>)</sup>	Insert first sample tube
REMOVE TUBE (Beep)	Remove tube

Continue to follow prompts. After all samples have been read, press STOP.

#### **Expected Results:**

- %CV (coefficient of variation) between standard duplicates of 10% or less.
- Absorbance reading for the 0 ppb standard should be between 0.8 and 2.0 for all assays.
- Correlation (r) of 0.990 or greater for all assays.
- Kit control within range specified on vial.
- Absorbance of negative control and standards should be as follows:

Negative Control>Std. 1>Std. 2>Std. 3.

- 3. Concentrations will be indicated for all samples on the RPA-I printout.
  - a) The concentration, as indicated on the printout, is multiplied by the appropriate dilution factor (if applicable) introduced in the procedure. The quantitation range of the kit is also multiplied by this factor.

EXAMPLE: Water samples were diluted 2-fold with methanol upon collection (see "Collect/Store the Sample" in this User's Guide). As a result, the concentrations listed on the printout should be multiplied by 2 to determine the sample concentration. The standard concentrations are also multiplied by 2 to give a quantitation range in water for this test kit of 0.5 to 10 ppb.

- b) Samples with an "nd" and no concentration listed have an absorbance greater than the negative control; therefore, no concentration can be computed for these samples. Results must be reported as < 0.5 ppb (or Standard 1 multiplied by the dilution factor.)
- c) Samples with an "nd" next to a listed concentration have an estimated concentration below the minimum detection level of the test kit. Results must be reported as <0.5 ppb (or Standard 1 multiplied by the dilution factor.)

NOTE: Any samples with concentrations determined to be lower than Standard 1 (the limit of quantitation) must be reported as < 0.5 (or Standard 1 multiplied by the dilution factor.) Quantitation is not possible below this standard as this is outside the linear range of the assay.

d) Similarly, samples with a "hi" next to a listed concentration have an estimated concentration higher than Standard 3 and must be reported as >10 ppb (or Standard 3 multiplied by the dilution factor.)

NOTE: In order to determine the concentration of samples with concentrations greater than Standard 3, they must be subjected to repeat testing using a diluted sample. A ten-fold or greater dilution of the sample is recommended with an appropriate amount of PCB diluent. This additional dilution must then be

taken into account when calculating the concentration. Please contact technical support for assistance in performing dilutions.

### Photometric Interpretation Using Other Photometers

Other photometers may also be used to interpret results obtained from the RPA-I photometer. It is important that the photometer be able to read absorbance at 450nm and that the instrument can read at a 1 mL fill volume. Absorbances obtained from other spectrophotometers (reading at 450 nm) may be used to manually calculate sample concentrations as outlined below.

- 1. Calculate the mean absorbance for each of the three standards and the negative control.
- 2. Determine the standard deviation and %CV (coefficient of variation) of each standard and ensure %CV is less than 10% for each.
- 3. Calculate the %B/Bo for each standard by dividing the mean absorbance value for the standard by the mean absorbance value for the negative control and multiplying the results by 100.
- 4. Construct a standard curve by plotting the %B/Bo for each standard on the vertical logit (y) axis versus the corresponding analyte concentration on the horizontal logarithmic (x) axis on the graph paper provided in the test kit. Graph papers are specific for each method. Use only the graph paper supplied with each kit.
- 5. Draw the best straight line through all points. Using the %B/Bo of the sample, the concentration can be interpolated from the standard curve.
- 6. Multiply results by the appropriate dilution factor (if applicable) introduced in the procedure. For example, if the sample was diluted 10-fold to increase the detection levels of the kit then the results must be multiplied by 10. This dilution also changes the range of the assay (standards) by the same factor.

NOTE: Do not forget to account for the 2x dilution introduced in the "Collect/Store the Sample" procedure for water samples.

### Limitations of the Procedure

The Rapid Assay® PCB Test Kit is a screening test **only**. Sampling error may significantly affect testing reliability. Adequate sample number and distribution are the responsibility of the analyst.

### **Ordering Information**

Description	Catalogue Number	
Rapid Assay® PCB Kit	A00133/A00134	
Rapid Assay® Accessory Kit**	6050100	
Adjustable Volume Pipet Tips (100-1000 uL)	A00013	
12.5 mL Combitip for Repeating Pipette (1 each)	A00009	
PCB Diluent A00136		
PCB Soil Proficiency Sample A00175		
Rapid Assay® Accessory Kit Rental 6997010		
** To obtain part numbers and pricing for individual items in the Accessory Kit contact SDI at the number below.		

### Ordering/Technical Assistance

Should you have any questions regarding this procedure prior to analysis contact Technical Service to avoid costly mistakes.

To Place an Order or Receive Technical Assistance, please call Strategic Diagnostics Inc. at:

Call toll-free 800-544-8881

Or 302-456-6789 Phone 302-456-6782 Fax

Web site: <a href="www.sdix.com">www.sdix.com</a>
E-mail: techservice@sdix.com

### **General Limited Warranty**

SDI's products are manufactured under strict quality control guidelines and are warranted to be free from defects in materials and workmanship. New instruments and related non-expendable items are warranted for one year from date of shipment against defective materials or workmanship under normal use and service.

Warranty obligation is limited to repair or replacement of the defective product or to refund of the purchase price, at the discretion of SDI. Other warranties, express or implied, are disclaimed. SDI's liability under any warranty claim shall not exceed the refund of the purchase price paid by the customer. Under no circumstances shall SDI be liable for special, indirect or consequential damages.

## Safety

To receive an MSDS for this product, visit our web site at www.sdix.com.

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Z00245.1, Rev 4/4/00

# Operation of the Repeater Pipet

### To Set or Adjust Volume

To determine the pipetting volume, the dial setting (1-5) is multiplied by the minimum pipetting volume of the tip (indicated on the side of the Combitip, e.g. 1~100 uL.)

### To Assemble Pipet Tip

Slide filling lever down until it stops. Then raise the locking clamp and insert the tip until it clicks into position. Be sure the tip plunger is fully inserted into the barrel before lowering the locking clamp to affix the tip in place.

### To Fill Tip

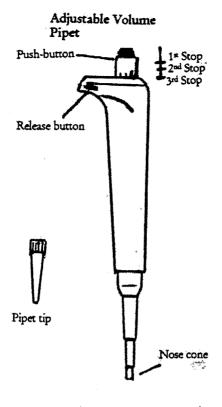
With tip mounted in position on pipet, immerse end of tip into solution. Slide filling lever upward slowly. Combitip will fill with liquid.

### To Dispense Sample

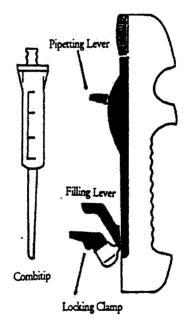
Check the volume selection dial to ensure pipetting volume. Place tip inside test tube so that tip touches the inner wall of tube. Completely depress the pipetting lever to deliver sample. NOTE: Dispense one portion of reagent back into the container to engage the ratchet mechanism and ensure accuracy.

### To Eject Tip

Empty tip of any remaining solution into appropriate container by pushing filling lever down. Raise locking clamp upward, and remove the Combitip.



Repeater Pipet



# Operation of the Adjustable Volume Pipet

### To Set or Adjust Volume

Press release button on side of pipette and turn the push-button to adjust volume up or down. Volume setting is displayed on top of pipet. See kit instructions for appropriate setting. Pipet will accurately dispense volumes between 100 and 1000 uL.

### To Assemble Pipet Tip

Gently push nose cone of pipet firmly into a pipet tip contained in the pipet tip rack.

### To Withdraw Sample

Keep pipet almost vertical. With tip mounted in position on pipet, press push-button to 1<sup>st</sup> stop and hold it. Place tip at bottom of liquid sample and slowly release push-button to withdraw measured sample. Ensure that no air bubbles exist in the pipette tip. If bubbles exist, dispense sample and re-withdraw. Slide tip out along the inside of the vessel.

### To Dispense Sample

Wipe any liquid from outside of tip taking care not to touch orifice. Place tip into tube, almost to the bottom, and slowly press push-button to 2<sup>nd</sup> stop. Hold push-button at 2<sup>nd</sup> stop when removing tip from tube.

### To Eject Tip

Press push-button to 3<sup>rd</sup> stop. Tip is ejected.

### STRATEGIC DIAGNOSTICS INC.

# RaPID Assay® PCB In Soil Application

### **Intended Use**

For detection of Polychlorinated Biphenyls (PCB's) (as Aroclor 1254) in soil. For testing in other matrices, please contact our technical support department at 1-800-544-8881.

### Materials Required but Not Provided

SDI Sample Extraction Kit

(Part Number: A00137EA/A00137EB)

### **Procedural Notes and Precautions**

- Prepare soil samples for analysis according to the procedure in the SDI Sample Extraction Kit Users Guide.
- After extraction and dilution of samples, follow the immunoassay procedure as described in the Rapid Assay ® PCB Test Kit User's Guide.
- The initial 2x dilution described for water samples in Step 1 of "Collect/Store the Sample" does not need to be performed for soil samples.

# **Quality Control**

A control solution at approximately 3 ppb (as Aroclor 1254) is provided with the PCB RaPID Assay® Kit. It is recommended that it be included in every run and treated in the same manner as unknown samples. If running standard soil procedures an acceptable result should be 2000 times the value stated on the control vial (i.e. 6.0 + or - 1.2 ppm) when the control results are corrected for the dilution factors (see Results section below).

## **Results Interpretation**

Interpret soil sample results as described in the RaPID Assay® PCB Test Kit procedure, accounting for the total dilution factor indicated in the table of the SDI Sample Extraction Kit Users Guide. Alternatively, program the

RPA-1 Analyzer as listed below to automatically correct for this dilution factor.

1. The RPA-I photometer (provided in the Rapid Assay® Accessory kit) can be used to calculate and store calibration curves. To obtain soil results from the PCB Rapid Assay® test kit on the RPA-I the following parameter settings are recommended:

Data Reduct:

Lin. Regression

Xformation:

Ln/LogitB

Read Mode:

Absorbance

Wavelength:

450 nm

Units

PPM

# Rgt Blk :

0

Calibrators:

# of Cals

4

# of Reps :

2

#### Concentrations:

#1 :

0.00 PPM

#2

0.50 PPM

#3

2.00 PPM

#4

10.00 PPM

Range

0.5 - 10.00

Correlation:

0.990

Rep. %CV:

10%

### Performance Data

The PCB RaPID Assay® does not differentiate between PCB and other related compounds. The table below shows compounds at the method detection limit (MDL) which is the lowest concentration of the compound in soil that can be picked up in the assay. The limit of

quantitation (LOQ) is an approximate concentration required to yield a positive result at the lowest standard, this is the lowest concentration of the compound in soil that can be quantified in the assay. The IC50 is the concentration in soil required to inhibit one half of the color produced by the negative control. It is also used to calculate cross-reactivity values to similar compounds.

Compound	MDL	LOQ	IC50
	(ppm)	(ppm)	(ppm)
Aroclor 1254	0.20	0.5	3.60
Aroclor 1260	0.20	0.3	2.30
Aroclor 1248	0.22	0.6	4.22
Aroclor 1242	0.34	1.2	8.80
Aroclor 1262	0.36	0.7	4.74
Aroclor 1232	0.84	2.6	18.76
Aroclor 1268	0.92	3.0	21.80
Aroclor 1016	0.94	3.6	25.60
Aroclor 1221	13.54	22.6	162.6

#### Soil Contaminants

Some contaminants found in soils that also contain PCB's can interfere with the analysis and cause false positives, false negatives or both when the compound is present at elevated concentrations. Interferences were assessed by adding increasing concentrations of some relevant contaminants to blank and PCB spiked soils prior to the extraction procedure. The concentration of the compound shown below produced no evidence of interference in a positive or negative direction in the 500 ppb to 10 ppm detection range of the procedure described above.

	concentration in soil oducing no interference
gasoline 25 transformer oil 5, 1-chloronapthalene 2, 1,2,4 trichlorobenzene 1,	0,000 ppm or 10% 5,000 ppm or 2.5% 000 ppm or 0.5% ,000 ppm or 0.2% 000 ppm or 0.1% ,000 ppm or 0.1%

If additional dilutions of the soil extract are made to detect soil PCB concentrations greater than 10 ppm, these interferences are diminished in direct proportions to the dilution made.

#### Range of Detection

The PCB RaPID Assay® has a range of detection in soil of 500 ppb to 10 ppm (as Aroclor 1254) when used in conjunction with the SDI Sample Extraction Kit.

### Recovery

PCB recoveries will vary depending on soil type, retention mechanism, solvent and extraction apparatus used, length of extraction period and levels of potentially interfering substances in the soil.

### STRATEGIC DIAGNOSTICS INC.

## Sample Extraction Kit User's Guide

### **Intended Use**

This extraction kit is for use with the appropriate immunoassay test kit. Each Sample Extraction kit contains the materials necessary to process twelve (12) soil or wipe samples.

### **Test Principles**

The reagents contained in the Sample Extraction kit have been optimized for fast, efficient removal of compounds from soil or surfaces and convenient preparation of the sample for immunoassay testing at levels of interest to the investigator. The system allows for reliable, convenient and cost effective determinations at the field testing or remediation site.

### **Performance Characteristics**

### **Precautions**

- Treat potentially contaminated samples as hazardous materials.
- Use gloves, proper protective clothing, and methods to contain and handle hazardous material where appropriate.
- Store all kit components at ambient temperature (18°C to 27°C or 64°F to 81°F).
- Do not mix reagents from kits of different lot numbers.
- When testing soil samples, samples obtained from areas adjacent to standing water, surface soils collected during or immediately after rain or snow, or any soils with relatively high amounts of water (≥ 30% by weight) should be dried prior to extraction. Contact technical service for recommended methods.
- Adequate sample number and distribution are the responsibility of the analyst.

- Do not dilute or adulterate test reagents; this may give inaccurate results.
- Cloudy or dark sample extracts may indicate the presence of interference in your sample. Please contact Technical Support if this occurs.

### **Materials Provided**

• Filter unit bottoms: 12 per kit

• Filter unit tops: 12 per kit

• Wooden spatulas: 12 per kit

Plastic Weigh Boats: 12 per kit

• Bulb Pipettes: 12 per kit

Ampule crackers: 12 per kit

Extraction jars: 12 per kit
 (Jars for soil extraction contain 3 ball bearings)

• 10 cm x 10 cm Plastic Wipe Templates: 12 per kit (PCB Wipe Kit only)

• Gauze Wipes: 12 per kit (PCB Wipe Kit only)

 Protective gloves: 24 per kit (PCB Wipe Kit only)

User's Guide

 Kit Specific Extraction Solution: 12 per kit as described below:

20 mL of 100% Methanol for use with:

Ensys PCB Soil/Wipe (Part #7020301/7021301) \*

Ensys Petro Soil (Part #7042301)

Ensys PAH Soil (Part #7061301) \*

Ensys Penta Soil (Part #7000301) \*

RaPID Assay PCB (Part # A00133/A00134) \*

RaPID Assay PAH (Part # A00156/A00157) \*

RaPID Assay CaPAH (Part # A00200/A00201) \*

RaPID Assay TNT (Part # A00186) \*

10 mL of 100% Methanol for use with:

Envirogard PAH in Soil (Part #7060600) \*
Envirogard BTEX in Soil (Part #7004000)
Envirogard TPH in Soil (Part #7042000)
Envirogard DDT in Soil (Part #7310000) \*
Envirogard PCB Soil/Wipe \*
(Part #7020800/7021600) or #7021500/7021600)

20 mL of 90% Methanol for use in:

Envirogard Chlordane in Soil (Part #7311000) Envirogard Toxaphene in Soil (Part #7420000) Envirogard Lindane in Soil (Part #7630000)

10 mL of 75% Methanol for use in:

Rapid Assay BTEX (Part # A00161/A00162)

20 mL of 75% Methanol with Sodium Hydroxide for use in:

Rapid Assay PCP (Part # A00110/A00111)

20 mL of 100% Methanol with Surfactant for use in:

Rapid Assay Cyclodienes (Part # A00216)

- \* Indicates extraction solution is also available in bulk (i.e. two 125 mL bottles per kit)
- Kit specific dilution material (for Ensys and RaPID Assay test kits)
  - For Ensys test kits dilution ampules will be provided based on customer specified detection levels.
  - For Rapid Assay test kits, 12 dilution vials with the appropriate volume of assay diluent (see "Dilute the Sample" section of this User's Guide) will be provided. The kit may also contain fixed volume disposable pipets and tips as appropriate.

# Materials Required and Ordered Separately

- 50 mL Combitips<sup>®</sup> for the Repeater pipettor for 1.0 mL to 5.0 mL dispensing volumes (if using bulk extraction solution)
- Eppendorf Repeater pipettor (if using bulk extraction solution)
- Portable balance capable of weighing 10 g (for soil samples)
- Electronic timer

**NOTE:** Order replacement Combitips® separately. See the "Ordering Information" section.

### Materials Required but Not Provided

- Protective clothing (e.g., latex gloves)
- Liquid and solid waste containers
- Marking pen

### Soil Procedure

### Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

- 1. Collect soil in appropriately sized and labeled containers.
- 2. Take care to remove excess twigs, organic matter, and rocks or pebbles from the soil sample to be tested.
- 3. Soils obtained from areas adjacent to standing water, surface soils collected during or immediately after rain or snow, or any soils with relatively high amounts of water (≥ 30% by weight) should be dried before testing. Contact Technical Services for recommended methods.
- 4. When comparing data from fields and laboratory methods it is important that split samples are obtained from thoroughly homogenized samples.
- 5. Store soil samples at 4°C (39°F), staying within the EPA recommended holding times for your analyte of interest.

### Weigh the Sample

- 1. Verify digital balance is calibrated correctly by pressing the ON/MEMORY button on the instrument and placing the 100 g weight (in the pocket of the instrument cover) onto the balance. If the instrument does not read 100±0.1 g, you must recalibrate the instrument as per the manufacturer's instructions provided with your accessory kit.
- 2. Place an unused plastic weigh boat on the digital balance (provided in the Field Accessory Kit).
- 3. Press the ON/MEMORY button on the digital balance. The balance will beep and display 0.0.

SDI Sample Extraction Kit

4. Weigh out  $10 \pm 0.1$  grams of sample into the weigh boat on the balance using a wooden spatula.

NOTE: If the balance turns off prior to completing the weighing of the sample, use an empty weigh boat to re-tare the instrument and then continue.

5. Repeat Steps 1-3 for each sample to be tested, using a new weigh boat and wooden spatula for each sample.

### **Extract the Soil**

- 1. Uncap an extraction jar (containing ball bearings) and place it on a flat surface. Label the extraction jar with the sample identification. Transfer 10 grams of sample from the weigh boat into the appropriately labeled extraction jar, using the same wooden spatula used to weigh the sample. Be careful to get your entire sample into the extraction jar.
- 2. Open the solvent ampule using the ampule cracker provided in your extraction kit by placing the ampule cracker over the scored neck of the ampule. The ampule cracker is designed to protect your hands from broken glass.
- 3. Pour the entire contents of one solvent ampule into the extraction jar and immediately recap the extraction jar. Do not leave the jar open or the solvent will evaporate and affect results.
- 4. Shake the jar vigorously for one full minute.
- 5. Allow the sample to settle for one minute or until a liquid solvent layer is observed above the sample.

NOTE: If the solvent layer is not observed within 15 minutes, contact Technical Support for assistance. Clay samples are often difficult to extract because they absorb the solvent. In this case, Technical Support may recommend decreasing the soil to solvent ratio. This will affect detection levels and should be discussed in advance

6. Repeat Steps 1-5 for each sample to be tested.

### Filter the Extract

 Insert the bulb pipet into the top (liquid) layer in the extraction jar (being careful not to disturb the lower, solid layer) and draw up some of the sample. Transfer at least ½ bulb capacity into the bottom

- portion of the filtration unit. Do not use more than one full bulb.
- 2. Press the top portion of the filtration unit (which is the piece with the cap and filter) into the bottom portion (containing the sample) until it snaps together or until the majority of the liquid has passed upward through the filter. Place on a flat surface.
- 3. Repeat Steps 1-2 for each sample to be tested.

NOTE: Do not store sample in the filtration unit for extended periods of time. The seal on the unit will not sufficiently prevent evaporative losses of the solvent. Evaporation of the solvent will affect results.

### Dilute the Sample

- I. Envirogard and Ensys test kits Use the filtered extract as "SAMPLE" in the test kit User's Guide procedure. The Ensys User's Guide describes a sample dilution method based on your individual testing needs.
- II. Rapid Assay Dilute the filtered extract into the appropriate sample diluent as described in the following table:

Kit	Extract Vol. (uL)	Diluent (mL)	*Total Dil. Factor	Test Range (ppm)
PCB	25	25	2000	0.5 to 10 (Aroclor 1254)
РАН	250	12.25	100	0.2 to 5 (Phenanthrene)
СаРАН	200	9.8	100	0.01 to 0.5 (Benzo(a)pyrene)
BTEX/ TPH	500	4.5	10	0.9 to 30 (total BTEX)
PCP	50	25	1000	0.1 to 10 (PCP)
TNT	50	25	1000	0.25 to 5 (TNT)
Cyclo- dienes	250	12.25	100	0.1 to 2 (Dieldrin)

\*Note: "Total dilution factor" takes the extraction dilution into account as well as the kit dilution (i.e. 10 g soil to 20 mL solvent is a 2x dilution).

a. Remove a pre-measured diluent vial from your extraction kit for each sample to be tested. Label vials

with the appropriate sample identification. Vials contain the volume of diluent specified in the preceding table corresponding to your test kit.

- b. Using the adjustable volume pipet (for volumes between 100 and 1000 uL) or the tan fixed volume pipet provided in the extraction kit (for 25 or 50 uL volumes) pipet the volume of filtered extract specified in the table above directly *into* the liquid in the corresponding diluent vial.
- c. Screw the cap tightly onto the diluent vial and mix by inverting several times. Repeat steps 1 and 2 for each sample being tested using a new, clean pipet tip for each one.
- d. The diluted extract should be used as "Sample" in the test kit User's guide procedure.

### Wipe Procedure

### Collect/Store the Sample

The following steps explain how to properly collect and store your samples.

- 1. Collect sample in appropriately sized and labeled containers.
- 2. Wearing a clean pair of protective gloves provided in the extraction kit, uncap an extraction jar.
- 3. Open the solvent ampule using the ampule cracker provided in your extraction kit by placing the ampule cracker over the scored neck of the ampule. The ampule cracker is designed to protect your hands from broken glass.
- 4. Pour the entire contents of one solvent ampule into the extraction jar and immediately recap the extraction jar. Do not leave the jar open or the solvent will evaporate and affect results.
- 5. Soak a gauze pad in the extraction jar containing solvent. Remove the gauze wipe from the extraction jar carefully squeezing the excess solvent from the pad back into the extraction jar.
- 6. Hold a clean 10 x 10 plastic template on the surface to be wiped. Wipe the entire exposed area according to proper wipe sampling techniques. The wipe should be damp when finished.

- Place the wipe back into the same extraction jar used in Step 4 and cap tightly.
- 8. Remove and discard the gloves.
- 9. Repeat Steps 1-7 for each sample to be tested.
- 10. Store samples at 4°C (39°F), staying within the EPA recommended holding times for your analyte of interest.

### Extract the Sample

- 1. Shake the jar vigorously for one full minute.
- 2. Repeat for each sample to be tested.

#### Filter the Extract

- 1. Insert the bulb pipet into the top (liquid) layer in the extraction jar and draw up some of the sample. Transfer at least ½ bulb capacity into the bottom portion of the filtration unit. Do not use more than one full bulb.
- 2. Press the top portion of the filtration unit (which is the piece with the cap and filter) into the bottom portion (containing the sample) until it snaps together. Place on a flat surface.
- 3. Repeat Steps 1-2 for each sample to be tested.

NOTE: Do not store sample in the filtration unit for extended periods of time. The seal on the unit will not sufficiently prevent evaporative losses of the solvent. Evaporation of the solvent will affect results.

### Dilute the Sample

- I. Envirogard and Ensys test kits Use the filtered extract as "SAMPLE" in the test kit User's Guide procedure. The Ensys User's Guide describes a sample dilution method based on your individual testing needs.
- II. Rapid Assay Dilute the filtered extract into the appropriate sample diluent as described below.

Kit	Extract Vol. (uL)	Diluent (mL)	Total Dil. Factor	Test Range (ppm)
PCB	25	25	2000	5 to 100 ug/100 cm2 (Aroclor 1254)

\*Note: "Total dilution factor" takes the extraction dilution into account as well as the kit dilution (i.e. 10 g soil to 20 mL solvent is a 2x dilution).

- a. Remove a pre-measured diluent vial from your extraction kit for each sample to be tested. Label vials with the appropriate sample identification. Vials contain the volume of diluent specified in the table above corresponding to your test kit.
- b. Using the adjustable volume pipet (for volumes between 100 and 1000 uL) or the tan fixed volume pipet provided in the extraction kit (for 25 or 50 uL volumes) pipet the volume of filtered extract specified in the table above directly *into* the liquid in the corresponding diluent vial.
- c. Screw the cap tightly onto the diluent vial and mix by inverting several times. Repeat steps 1 and 2 for each sample being tested using a new, clean pipet tip for each.
- d. The diluted extract should be used as "Sample" in the test kit User's guide procedure.

### Limitations of the Procedure.

Sampling error may significantly affect testing reliability. The distribution of contaminants in soils can be extremely heterogeneous. Adequate sample number and distribution are the responsibility of the analyst.

# **Ordering Information**

Description Catalogue Number		
SDI Sample Extraction Kit (with methanol in ampules or bulk)	Contact Customer Support	
50 mL Combitip for Repeating Pipette (1 each) 6005600		
Portable balance**	A00131	
Eppendorf Repeater Pipettor** A00008		
Electronic Timer** A00015		
** These items are also included in our field accessory kits which are available for rent or purchase.		

### Ordering/Technical Assistance

Should you have any questions regarding this procedure prior to analysis contact Technical Service to avoid costly mistakes.

To Place an Order or Receive Technical Assistance, please call Strategic Diagnostics Inc. at:

Call toll-free 800-544-8881

Or 302-456-6789 Phone 302-456-6782 Fax

web site: <a href="www.sdix.com">www.sdix.com</a>
e-mail: <a href="techservice@sdix.com">techservice@sdix.com</a>

### **General Limited Warranty**

SDI's products are manufactured under strict quality control guidelines and are warranted to be free from defects in materials and workmanship. New instruments and related non-expendable items are warranted for one year from date of shipment against defective materials or workmanship under normal use and service.

Warranty obligation is limited to repair or replacement of the defective product or to refund of the purchase price, at the discretion of SDI. Other warranties, express or implied, are disclaimed. SDI's liability under any warranty claim shall not exceed the refund of the purchase price paid by the customer. Under no circumstances shall SDI be liable for special, indirect or consequential damages.

## Safety

To receive an MSDS for this product, visit our web site at www.sdix.com.

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# ATTACHMENT 4 RESPONSE TO AGENCY COMMENTS

# Response to Agency Comments on the Draft Upstream Patrick Bayou Characterization Sampling and Analysis Plan

Comment	Section/Page Number	Agency/Comment	Response
1	General	<b>TCEQ/</b> It was identified in the SAP that the COPCs at the Site have historically accumulated in soft sediments. In addition, it is identified that the grab sediment samples will be collected in areas identified from sediment probing in upstream areas of the Site between PB066 and PB101. If preferred sediment sample conditions (soft sediment accumulations that have a thickness of 10 cm or more) cannot be found throughout the areas of the Site between PB066 and PB101 will the sampling crews still collect sediment samples throughout the areas of PB066 and PB101 to characterize the COPCs in the sediments of the upstream areas of the Site?	If areas of accumulation of shallow soft sediments (less than 10 cm depth) are present, the field team will attempt to sample these areas.
2	General	<b>TCEQ/</b> It is identified in the SAP that up to 36 sediment grab samples will be collected. What will be the minimum number of sediment grab samples collected? Will the minimum number of samples be able to characterize the distribution of the COPCs in the sediments?	The minimum number of sediment grab samples will be set according to the extent of soft sediment coverage observed between PB066 and PB101. It is not possible to pre-determine the minimum number of samples.
3	General	<b>TCEQ/</b> It is identified in the SAP that the results of the immunoassay testing will be used to identify specific samples for additional laboratory analysis (PAHs and PCB Aroclors) of the grab sample sediments. Why will only PAHs and PCB Aroclors be analyzed? Will there be future testing on the samples for additional COPCs?	The 2009 sediment sampling of sites above PB076 showed relatively low levels of mercury (less than 0.05 mg/kg), Sum DDx (less than 3 ug/kg), and Total Dioxin/Furans (less than 10 ug/kg). The 2009 sampling did indicate a higher concentration of Total PAH and Total PCB Congeners at PB081 than samples from earlier sampling events (2000 to 2006). Therefore, the analytes for the soft sediment samples sent to the laboratory will be PAHs and PCB Aroclors. No additional testing or future testing for these samples is planned.
4	General	<b>TCEQ/</b> It is identified in the SAP that four surface water samples will be collected from the upstream portion of Patrick Bayou between stations PB066 and PB101. Will the sample size be able to characterize the distribution of the COPCs in the surface water?	This data will extend the 2009 surface water sampling at PB059 and PB076 to characterize the possible distribution of PCB congeners in the surface water. Four samples are planned – PB066, PB101, and two samples to be determined after the field testing of sediments is completed and will focus on areas with elevated Total PCBs in the soft sediments. This level of sampling density longitudinally is higher than in the 2009 effort and should provide adequate characterization in the upper portion of the bayou. DDTs were deleted from surface water COPCs in the Draft Baseline Ecological Risk Assessment Work Plan and therefore will not be analyzed for in this surface water collection.
5	1.0 Introduction and Purpose	<b>TCEQ/</b> This SAP describes the rationale, objectives, study design, and methods for the characterization of the sediments in the upstream portion of the Patrick Bayou Superfund Site (Site; Stations PB066 to PB101), and culverts that run underneath State Highway (SH) 225, just upstream of the Site boundary. Are there any plans to sample the underground portion of the bayou between SH 225 and PB101? Why or why not? If not, are there any reasons that may trigger sampling at these "more" upstream areas?	At this time, there are no plans to sample the underground portions of the bayou between SH225 and PB101 because of health and safety concerns. We are sampling at the culverts at SH225 at the upstream end of the underground portion. This is the only place of significant accumulation of soft sediments that have been identified based on previous reconnaissance by Lubrizol. Because of confined space entry and related health and safety concerns, we do not intend to sample further into the culverts at this time. If significant accumulation of soft sediments are observed at the entry and exit of the underground portions of the bayou during the sampling event, the field team will note the existence and develop a plan in the future, if deemed necessary, to characterize the sediments in this portion of the bayou.
6	2.0 Previous Results, Study Objectives, and Design	<b>TCEQ/</b> Upon review of the May 2010 sediment and surface water report, it appears that the sediment data in Appendix A1 lacks sample results for stations upstream of PB037. This appendix (in the 2010 report) should be revised to reflect the entire data set for the data from samples collected in 2009.	This has been addressed and corrected in the May 2010 sediment and surface water report.
7	2.0 Previous Results, Study Objectives, and Design	<b>TCEQ/</b> It appears that the units for the PCBs in surface water should be ng/L rather than ug/L (first full paragraph following the bullets on pg. 3)	This is correct and the SAP will be corrected.
8	3.1.1 Sediment Probing Sample Locations and Frequency – Station PB066 to Station PB101 and 3.22 Target Analyte List – Box	<b>TCEQ/</b> According to the SAP, up to 36 grab samples will be collected from identified soft sediment accumulations that have thickness of 10 cm or more, to determine the surficial concentrations of total PCBs using immunoassay test kits on-site. Each sample will be split, and six of the split samples will be submitted for off-site laboratory analysis of PCB Aroclors and PAHs. In contrast, the sediment grab samples and core collected from the culverts at SH 225 will be analyzed for PCB congeners. If the PCB analyses differ, how can the JDG reliably compare the	Direct comparison between upstream culvert sampling and lower gunite channel samples is not a specific objective; only relative levels of Total PCBs will be compared. The culvert sampling is designed to provide additional information on potential off-site upstream loadings, while the sediment sampling between PB066 and PB101 (lower gunite channel) is to characterize potential areas of localized PCB

# Response to Agency Comments on the Draft Upstream Patrick Bayou Characterization Sampling and Analysis Plan

Comment	Section/Page Number	Agency/Comment	Response
	Culverts	downstream/upstream results?	and PAHs in this area. In the lower gunite channel, the immunoassay approach will yield rapid, efficient information to characterize distribution of PCBs and PAHs in surface sediments; laboratory analysis of samples from this area are for the explicit purpose of confirming results from the immunoassay field test. As the immunoassay is calibrated to an Aroclor standard, it is appropriate to utilize Aroclor analysis for confirmatory laboratory analysis. For the upstream loading samples, use of PCB congeners allows for comparability with previous sampling and ensures adequate detection sensitivity.
9	3.2 Experimental Design – Box Culvert Sediment Sampling	<b>TCEQ/</b> According to the SAP, sediment samples will be collected at the upstream end of the box culverts that run underneath SH 225. TCEQ suggests that the JDG also collected sediment samples from the downstream end of each culvert. These samples should all be analyzed for the same analytes planned for the upstream end. These results should indicate if any of the specific culverts are problematic and need further evaluation.	The upstream culverts were chosen for sampling because this is where significant soft sediment accumulation has been observed previously. If any accessible soft sediment accumulations at the downstream end of the culvert exist, a grab sample of those sediments will be collected; however, the hydrodynamic regime in this area is very high and we do not expect an accumulation of soft sediments at the downstream end of the culvert.
10	3.2.1 Sample Locations and Frequency – Box Culverts	TCEQ/ Will the four culvert samples be processed as discrete samples or will they be composited.	The culvert samples will be processed as discrete samples.
11	5.6.1 Sediment Probing and 5.6.2 Surface Sediment Sampling	<b>TCEQ/</b> According to the SAP, up to 36 grab samples will be collected and tested where soft sediment accumulation thicknesses are 10 cm or greater. Bank sediments should be avoided when probing for sediments to sample. If the sampling personnel only probe from the shoreline, it is likely that only back sediments will be encountered. The bank sediments are not depositional.	We agree.
12	Section 2.2	<b>TCEQ/</b> Since the sampling and analysis plan (SAP) is "an extension of the 2009 evaluation" (Sec. 2.2 Subject Report) it is, similarly, presumed to contribute to the modeling effort at Patrick Bayou. As such, it is recommended that the section be augmented with a brief description of how these data will supplement the modeling effort	We do not anticipate that these data will be used in the modeling, but instead will focus on identifying locations of elevated PAH and Total PCB concentrations in the upper portion of the bayou that may act as a source area.
13	Section 3.1.2	<b>TCEQ/</b> Based on immunoassay on-site tests, grab sample splits will be selected and sent to lab. On what immunoassay test aspect will such selection be based?	Samples that show a range of relatively elevated Total PCB concentration will be selected for lab analysis.
14	Section 3.1.2	<b>TCEQ/</b> The number of splits and/or grab samples actually obtained may be adjusted in the field "as needed" (pg. 8, Subject Report). What are example contingencies that would force an adjustment? Is a downward adjustment possible?	Only if the extent and existence of soft sediments is considerably limited between PB066 and PB101 (which is not expected, based on previous sampling events) would less than six samples be sent to the off-site lab for analysis. Upward adjustments may be considered based on field results and observations from the field team.
15	Section 5.6.2	TCEQ/ See comment 13 and 14 above and revise accordingly.	The SAP will be revised to reflect our responses to comments on items #13 and #14 above.
16	Section 5.6.2.2	<b>TCEQ/</b> The concentrations of the PCB immunoassay tests will be recorded on an "analytical bench sheet." However, it does not appear that the "analytical bench sheet" is included in the Field Forms to be completed in the subsequent report (Attachment 2, Subject Report). Such data should be included in the associated report.	Agreed. An analytical bench sheet will be included in the SAP.
17	Section 5.6.2.2	TCEQ/ The anticipated effected detection limit(s) of the PCB immunoassay tests should be included in the subject report – and "analytical bench sheet."	Agreed. The detection limit of the RaPID Assay Test Kit for Total PCBs is 0.5 ppm (as Aroclor 1254). This limit will be stated in the SAP and on the analytical bench sheet.
18	General Comment	<b>USEPA/</b> This plan focuses on the potential contribution to Patrick Bayou from upstream sources. The possibility (though not high) that some tidal influence may have actually moved some small amount of Patrick Bayou COPCs upstream should be considered and discussed.	Agreed. The following text will be added to Section 2.1; "Although Patrick Bayou is a tidally influenced system, upstream areas (i.e. upstream of Station PB076) are generally not influenced by tides and flow is generally downstream. However, it should be noted that it is possible that tidal flow reversals may occur on an infrequent basis and thereby act as a source to upstream sediments as well."
19	Section 5.6.2.2	<b>USEPA/</b> Suggest including information/steps on calibration and more details on the standards. Also recommend providing information related to this methods accuracy and precision. Finally, please provide some citations where this has been used.	The RaPID Assay Test for PCBs User's Guide has been referenced in the SAP text and added to the document as Attachment 3. Text has been added to the SAP in Section 5.6.2.2 to address the use of this method and accuracy and precision.